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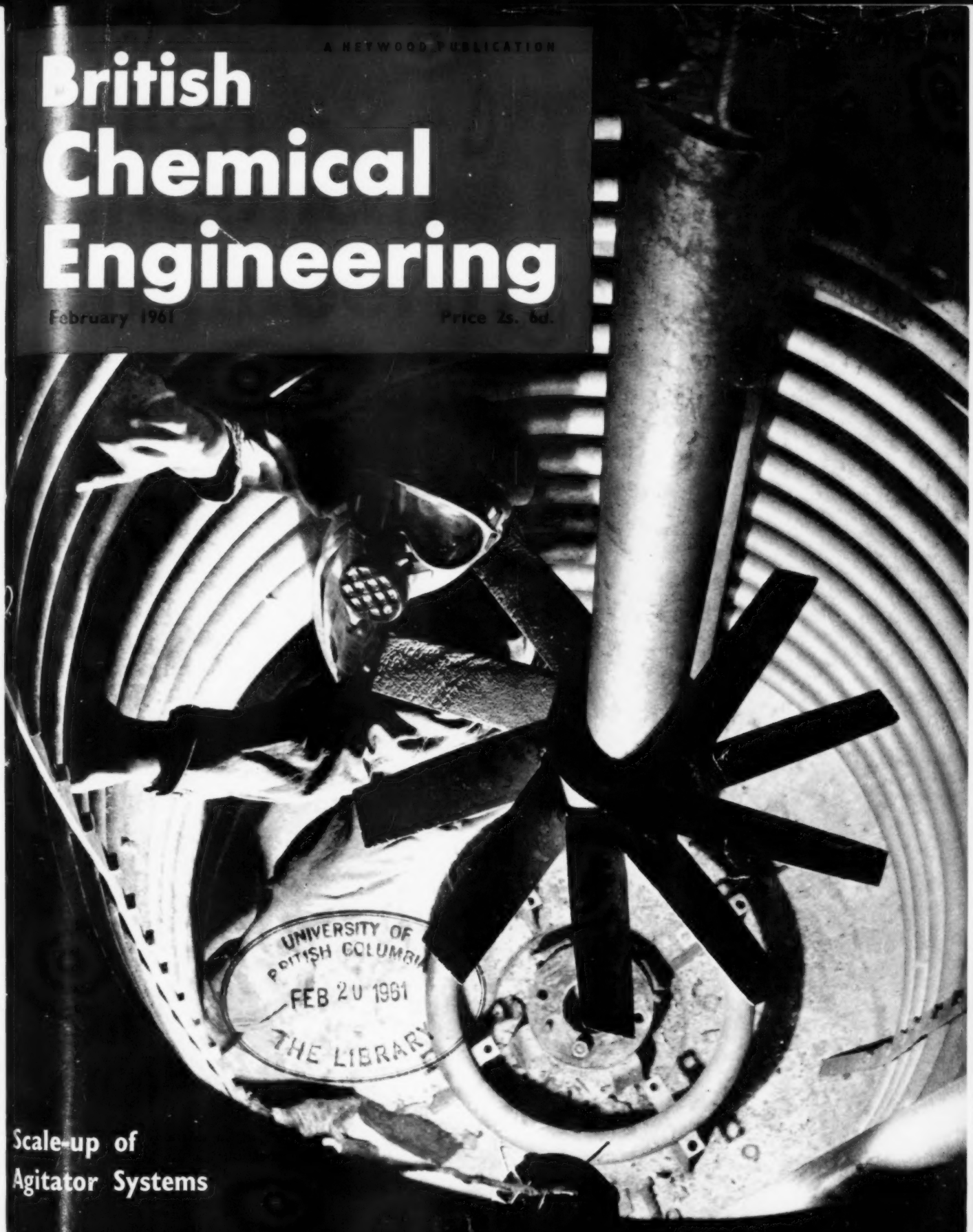

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## Scale-up of Agitator Systems

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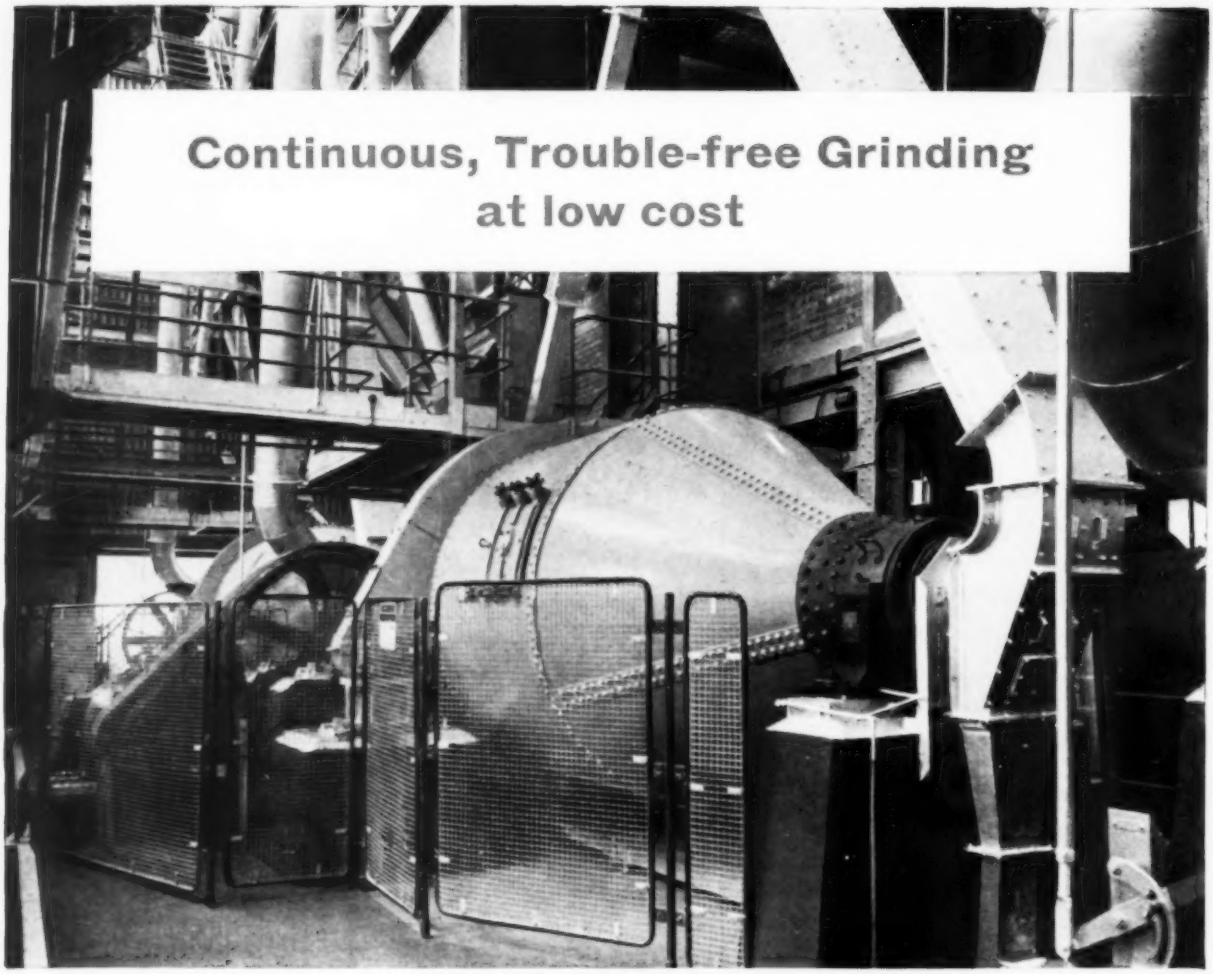


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# DIGEST OF INDUSTRIAL AND TECHNICAL DEVELOPMENTS

## Resisting Hot Hydrochloric Acid

FINDING a suitable corrosion-resisting material for hydrochloric acid at high pressures and temperatures up to the boiling point is something of a problem; which makes all the more interesting a recent solution tried at a Mexican refinery (Petroleos Mexicanos refinery, Atzcapotzalco, Mexico).

The particular vessel for use in an isomerization process in this refinery weighs some 13,000 lb and operates at 350 psi and at 230°C. Its 7/8-in. steel shell was lined with 7/64-in. sheets of 65 per cent Ni, 30 per cent Mo, 5 per cent Fe alloy (one of the Hastelloy group of alloys). The main vessel shell was welded by the metallic-arc process with coated stainless steel electrodes for the carbon steel to alloy welds, a similar though more complex procedure being used for lining and the welding of the elliptical head. The spaces between adjacent liner sheets were covered by two-inch wide, 14 gauge strips of the alloy which were welded to the base sheets by the inert-gas, metallic-arc welding process using bare wire of the alloy as the filler metal.

The liners were tested by applying pressure behind the overlay strips, for which purpose air at 15 psig was introduced into an overlay-strip weep hole at one end of the vessel while all the other weep holes which were provided under the overlay strips were checked for air flow, and then plugged. The interior seams were checked for leakage by preliminary soap and water tests, followed by a much more sensitive test using fluoro-carbon refrigerant vapour and a leak detector. The plugs were then removed and an internal halogen test carried out on all the external seams and weep holes. Finally, the weep holes in the bottom head were plug welded, the bottom head jacket was installed, and the entire unit prepared for the final hydrostatic testing; this was performed at 525 psig in the shell and 115 psig in the jacket.

The pressure vessel, which cost U.S. $22,000, successfully passed these tests and is now in operation.

## Temperature Effects in Magnetic Separation

AS the third main separation stage in the treatment of some New South Wales beach sands, one fraction is magnetically fractionated. Laboratory results however have indicated full scale plant requirements that are only three quarters of those actually in use. L. T. MORGAN has recently indicated how he followed up the possibility that the difference between the two cases was essentially one based on differences of operating temperature. Laboratory samples are introduced cold into a cold machine; feed to the production machine is around 70-90°C. The hypothesis of temperature effect was confirmed on preliminary tests which showed that extraction in the laboratory magnetic separator at 100-120°C gave an extraction of roughly half that obtained with the same sample at ambient temperature. Systematic investigation of these effects using controlled mixtures of four magnetic fractions of varying susceptibility made it possible to construct curves of per cent extraction against temperature. Mr. MORGAN, reporting this work*, has also discussed why this heat effect has not been recognised. Possible reasons are that each operating company has had its own particular set of conditions at the point of primary magnetic separation, and sufficient magnetic separation capacity has been installed to meet that case. Operators using belt type or glass plate carrier type for the primary separators will have taken precautions for cooling to protect the rubber or the glass; they will thus have helped to lift efficiency of their separation.

This loss of susceptibility with temperature was found to apply only to the ferro-magnetics present in the beach sands, and has no application to the slightly magnetic minerals either conducting or non-conducting in character.

* *Mining and Chemical Engineering Review* Vol. 52, No. 11, p. 70.

## Butane Freezing for Extracting Fresh Water from the Sea

FOR the conversion of saline water to fresh water, a pilot plant utilising butane as a refrigerant is under construction by the Blaw-Knox Company of America. At the recent 53rd Annual meeting of the American Institute of Chemical Engineers, MR. GEORGE KARNOFSKY of the Blaw-Knox Company gave some details of the new process.

Sea water is partially frozen to a slush by direct contact with boiling butane at a pressure slightly below atmospheric. Since butane (b.p. 31°F at atmospheric pressure) will boil below the freezing point of salt water, the heat of vaporisation (165 Btu/lb of butane) is supplied by the heat of freezing of water (144 Btu/lb of water). It is necessary only to bring butane and water into intimate contact.

The slush is washed free of salt with part of the product water. Then the washed ice is melted by the reverse of the freezing process. Butane vapour from the freezer is compressed to slightly above atmospheric pressure, where its condensation temperature is above 32°F, and brought into intimate contact with the ice. Ice melts and butane condenses, to be recycled to the freezer. Product water and reject concentrated sea water leave through heat interchangers countercurrently to the feed sea water.

Because there is direct heat transfer in both the freezing and melting steps, and because the depression of freezing point of partly concentrated sea water is small, little energy is expended in this heat pump cycle. The energy cost is estimated at only 26 kWh/1000 gallons of product water, and equipment costs are minimised. It should be possible to build a plant with a capacity of 10 million gallons per day for a cost of $20 million; and to produce water at a cost for power and chemicals of 19 cents per gallon.

## The Economics of Fractional Solidification

SOME cost estimates on the commercial possibilities of refining naphthalene by fractional solidification have been made in the course of a detailed theoretical and experimental study of fractional crystallisation by zone melting of 10 per cent mixtures of $\beta$-naphthol and of benzoic acid in naphthalene, by William R. Wilcox of the Lawrence Radiation Laboratory, University of California*.

In the cost estimates, the starting material was assumed to be 500,000 lb per year of 1 per cent by weight $\beta$-naphthol in naphthalene, and the products 0.1 per cent by weight $\beta$-naphthol in naphthalene and 30 per cent by weight $\beta$-naphthol in naphthalene.

For a large scale separation such as this, the conventional batch-type zone-melting process is generally impractical. Instead, therefore, a zone-void refiner was considered, which provides a multistage semicontinuous operation. The calculations indicated that the total length of refiner needed to process 500 lb. per year of crude naphthalene would be 1,100 ft—possibly using 56 refiners each 20 ft long. The summarised costs are given below:

| | |
|---|---|
| Installed cost of one refiner ... | 24,100 dollars |
| Total installed equipment cost ... | 1,366,410 dollars |
| Total physical cost ... ... | 2,190,000 dollars |
| Total project cost ... ... | 3,940,000 dollars |
| Total capital necessary ... ... | 4,085,250 dollars |
| Annual cost ... ... ... | 1,380,000 dollars |
| Cost per lb of crude naphthalene | 2.76 dollars |

Thus, zone melting cannot be considered an economic process for the bulk separation of cheap organic materials. However, for small-lot purification of some chemicals, it would be competitive.

Moreover, the cost is roughly proportional to boundary layer thickness. Possibilities suggested for reducing the thickness of the boundary layer are (a) the use of highly conductive materials running through the zone—already proved to be beneficial in the experimental investigation; (b) rotation of the refiner tube, to enhance free convection effects by centrifugal force; (c) vibration of the tube, perhaps ultrasonically. These two latter suggestions have not yet been tried experimentally and require further investigation.

* UCRL-9213. Lawrence Radiation Laboratory, Berkeley, California. June 1960.

## Industrial Non-Porous Graphite

A NEW type of graphite of greatly reduced permeability, with many potential applications in the chemical and metallurgical industries, has been developed by the Hawker Siddeley Nuclear Power Company. Improved types of graphite were required for use as fuel element cases in high-temperature gas cooled reactors, and to meet this specification the Hawker Siddeley company developed the R.A.E. furfuryl alcohol process from a laboratory experiment to the production stage. The material thus produced not only satisfied the permeability requirements but also showed marked improvements in mechanical and physical properties as compared with standard graphites.

Subsequently the company have introduced further processing developments designed to extend the fields of application of the new material. They are now able to offer erosion-resistant coated grades suitable for rocket nozzle throats; an impregnated grade in the form of seals and gland packings for use in pumps; impregnated graphite bursting discs; graphite for high temperature bearings and bushes; corrosion resistant grades for chemical plant and heat exchangers; oxidation resistant graphites; and a type specially suited to the jigs used in transistor component manufacture.

The treated graphite is capable of holding a vacuum of 10 microns whilst heated at the same time to 1,800°C., under inert gas conditions. Mechanical and physical properties compared with those of standard graphite are given in the Table below.

| | *Standard Graphite* | *Treated Graphite* |
|---|---|---|
| Flexural strength, lb per sq. in. ... ... ... | 3600 | 5700 |
| Elastic modulus, lb per sq. in. ... ... ... | $1.8 \times 10^6$ | $3.0 \times 10^6$ |
| Electrical resistivity, milliohm cm. ... ... | 1.9 | 1.5 |
| Thermal expansion per deg. C. cm. ... ... | | |
| 300°C | 2.0 x 10-6 | 2.1 x 10-6 |
| 600°C | 2.2 x 10-6 | 2.7 x 10-6 |
| 900°C | 2.6 x 10-6 | 3.0 x 10-6 |
| Ultimate tensile strength, lb per sq. in. ... ... | 3200 | 5900 |

New plant is being installed to produce treated graphite at the Langley works of the company. At present, one carbonising and one graphitising furnace are installed, but eventually it is planned to have four carbonising and two graphitising furnaces in a semi-circular layout. The 1,000°C. controlled atmosphere carbonising furnace has a charge capacity of 4 ft 6 in. diameter by 14 ft 6 in. long Temperature control within very fine limits is automatically programmed from a control panel. The 3,000°C. controlled atmosphere graphitising furnace, which can be operated under a vacuum of 0.1 mm up to temperatures of 1,700°C., is said to be four times larger than any other graphitising furnace and can accommodate graphite components up to 10 ft long. To withstand the high temperatures, the graphitising furnace is itself graphite lined.

## Samuel Clegg

IN the applied chemistry of gas manufacture SAMUEL CLEGG, who died 100 years ago on January 8, was an engineer ever designing plant for producing and purifying coal-gas rather than chemist trying out processes from the laboratory scale. As a Mancheter man and pupil of DALTON he was schooled in the chemistry of the late 18th century. Yet he goes down in chemical engineering as one who began coal carbonisation in thin layers in continuous retorts at a time when the fixed horizontal fireclay retort was regarded as all that was necessary. CLEGG became assistant to BOULTON and WATT in early gas-manufacture, his work with MURDOCH being a foundation for his future role as the first true gas engineer.

CLEGG soon broke away from the moderate progress possible with the Soho Foundry partners where gas-lighting was concerned. He had installed a lime purifying-apparatus at Stonyhurst College in 1811; hence he took to an independent practice in 1813 by coming to London and very quickly making his name by installing a gas-producing plant at Ackermann's publishing shop in the Strand. Soon perceiving the disadvantages of paying for gas on a time basis, he strove to invent a dry meter, but had to be satisfied with a water or wet meter in 1815, this being followed by a second type with revolving drum. CLEGG introduced various forms of burners including a variation of the Argand burner invented by the Geneva scientist; but far more important were his larger inventions seeking to improve the carbonisation process. In 1815 he patented a type of continuous retort in which coal was carbonised in shallow trays fixed to radial arms rotating around a central axis, the coal in thin layers being

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charged at the cool side of the retort and passed to the hot zone. A second form known as a "web" retort used an endless chain to carry the coal to the hot zone and discharge the coke. Although others had experimented with lime purification for removing "the Soho stink" due to hydrogen sulphide, CLEGG was a more practical man in outlook from 1809 when he tried his first wet lime purifying-plant.

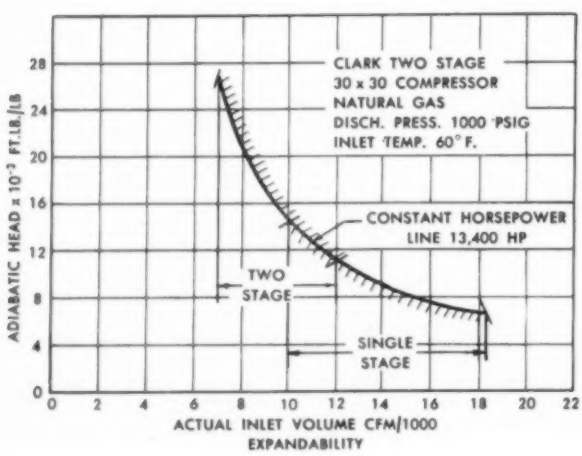
As chief engineer to the new Gas Light and Coke Company and consultant in later years, CLEGG was a leader in the industry despite an intermediate period when he lost a fortune in a Liverpool engineering venture. He supervised the laying of miles of gas mains, devised the hydraulic main in gas works and gas-holders, all before publishing a Practical Treatise on Coal-gas Manufacture.

## Two-Stage Centrifugal Pipeline Compressors

CENTRIFUGAL pipeline compressors for the transportation of natural gas have proved reliable and economical in operation. With a trend towards larger pipe sizes and higher gas pressures, more powerful drive units can be foreseen which in turn require still higher compression ratios. Already, with the availability of 15,000 h.p. drive units, a compression ratio of 1.5, corresponding to a comparatively high head of about 18,000 ft at 60°F, is required for the low-capacity condition. If this ratio is obtained in a single impeller, it will adversely affect performance: it could result in lower-than-optimum efficiency, a flatter head-capacity curve or a reduced stable range of operation.

One solution to this problem put forward by Clark Bros. Company, Olean, New York, is a two stage compressor in which the total compression ratio of 1.5 is equally divided between the two impellers. The blades are backward-bent approximately 35°. The specific speeds of first and second stage impellers are respectively 74 and 69, as against 43 for a single-stage design with a correspondingly lower efficiency. The backward-bent blades give a continuously drooping head-capacity curve which will permit considerable pressure fluctuations without developing surge.

The Clark two-stage compressor is of the "barrel" type which has proved very successful in the high-pressure-low molecular-weight field. Pressure loading within the machine is essentially equal in all directions. The shaft, although relatively long, is of the "stiff" type. The first critical speed of the rotor is well above maximum running speed. The two shrouded impellers are mounted back to back, reducing the net hydraulic thrust to the minimum. The two-stage arrangement can be converted to a single stage by merely replacing the internal "bundle" with internals designed for single stage operation, identical seals, bearings and shaft being retained.




The two stage arrangement employs impellers with different design points, sized to perform at optimum efficiencies based on their respective Q/N values. The performance of both impellers can be modified by the use of inlet guide vanes, over a flow range approximately ± 10 per cent. The expandibility of the two-stage compressor is demonstrated in the graph showing the estimated ranges of performance for a single and two-stage operation. At constant horsepower, change in capacity from maximum is 2.6 to 1 while still maintaining 80 per cent polytropic efficiency. Some further expansion is possible with correspondingly lower efficiency.

Four Clark 30 x 30 two-stage compressors of the type described, developing a total compression ratio of 1.5 and absorbing 13,400 h.p. at 100 lb per sq in. gauge discharge pressure have now been operating satisfactorily since October 1958.

## Red Iron Oxide Manufacture by Fluidisation

THE possibilities of fluidisation techniques for the manufacture of red iron oxide pigment from ferrous sulphate have been investigated at the Research Institute for Chemical Heavy Industry, Hungary. The experimental process, as described by TIBOR BLICKLE*, comprises three stages:

(1) Dehydration, oxidation and roasting of the ferrous sulphate using fluidisation equipment;
(2) Washing the raw pigment by fluidisation with water;
(3) Purifying the effluent gases by foam columns.

In the experiments, Stage 1—dehydration, oxidation and roasting—was carried out in a three-layer reactor. The influence of dwelling times, layer temperature and air velocity on iron oxide content and colour of the pigment were investigated. The most favourable conditions were as follows: in the upper layer, temperature 200°C, gas flow rate 30-35 cm/sec. In the middle layer, with a temperature of 500°C, the ferrous sulphate is completely oxidised. Solid matter leaving this layer is completely free from water content.

From the viewpoint of pigment manufacturing, the optimum temperature of the lower layer is 700°C, with a dwell of 1 hour and a gas flow rate of 40 cm/sec. Under these conditions the discharged product contains 96 per cent iron oxide.

For washing by fluidisation, the best fluidised layer was developed when the average grain size of the iron oxide was below 60 microns. With increasing rate of fluid flow, the solution rate was correspondingly increased. Fluidisation proved to be especially favourable because the basic iron sulphate in the product—which dissolves only very slowly and forms a flocculent precipitate in the water—is carried off by the water due to its low specific weight. As an example of the advantage of fluidisation, for washing 100 kg of iron oxide by the conventional periodical sedimentation method, three vats of 80 cu m capacity each are needed, whereas a fluidisation washer of only 3 cu m is sufficient for the same task. For the sedimentation method 128 litres per kg of water are required as against 50 litres per kg for fluidisation washing. Moreover the latter process is continuous and easily mechanised.

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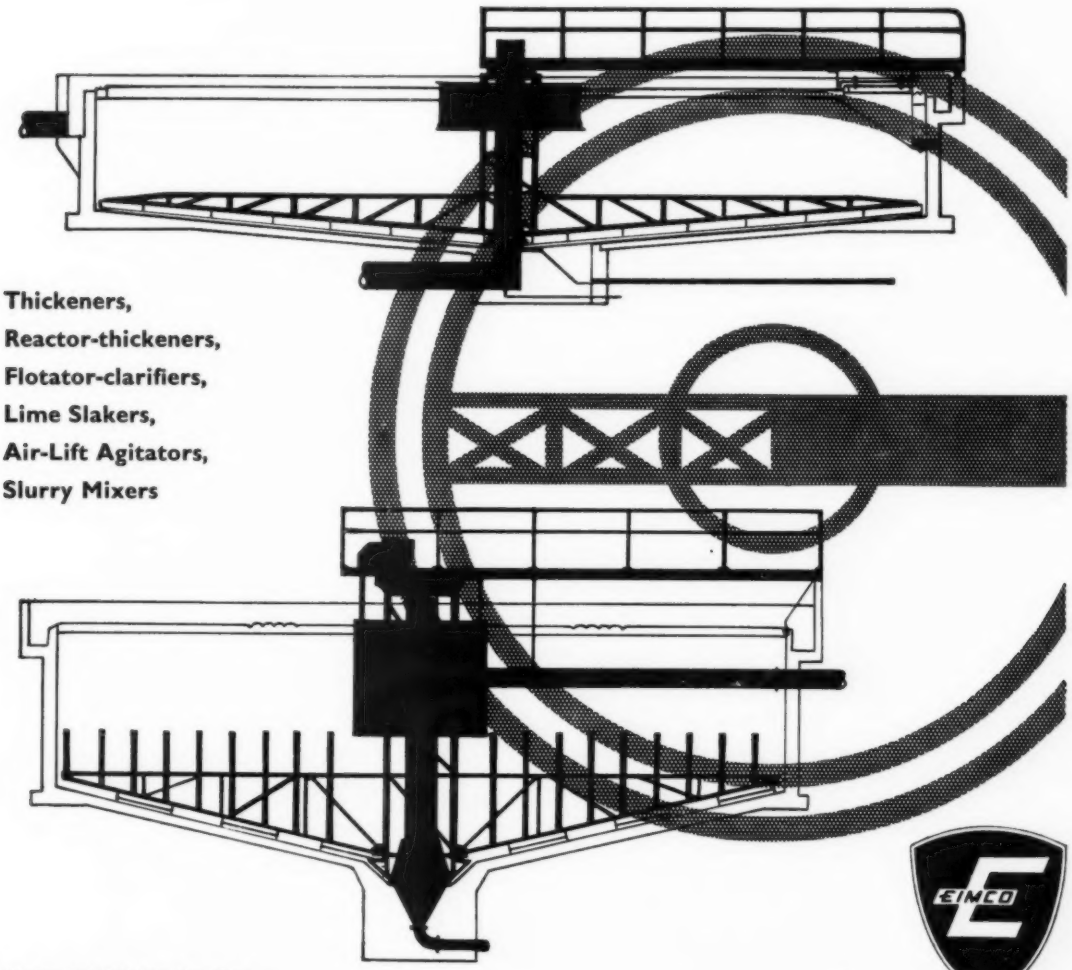

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Before discharging to atmosphere the gases are purged of $SO_2$ and $SO_3$ by passing through a foam column containing 8 per cent milk of lime. Alternatively, the gases can be used for sulphuric acid production, after filtering off dust and cooling in the foam column to below 20°C.

Preliminary experiments on the drying of pigment slurry by fluidisation have shown promise. When the slurry is fed continuously in little clusters or lumps into the fluidised layer, it agglomerates to form small granules and may be readily dried.

* Hungarian Heavy Industries, Summer 1960, page 27.

## Which Route for Ethylene Oxide?

THE rapid development of chemical engineering has enabled designers and constructors to solve problems in a number of processes which previously were in a state of enforced stagnation. In some cases, for a particular chemical, two routes were available, one of which remained undeveloped. Ethylene oxide is a case in point with its chlorhydrin and direct-oxidation routes, for though the direct-oxidation route was discovered by Lafort in the late 20s it is only very recently that it became competitive with the chlorhydrin process. According to R. P. VAN OOSTEN* the direct-oxidation method in present conditions is definitely to be preferred both in view of the higher purity of product and on economic grounds, even allowing for the caustic soda produced in the course of making the chlorine required by the chlorhydrin process. He draws attention to some variations of the oxidation process, for example, the source of oxygen may be either air, or oxygen from a gas separation plant, and some processes have attempted a fluid bed catalyst. Where capacities are below 6000 to 10,000 tons per year, processes relying upon air directly as the oxygen source, are the more economic.

VAN OOSTEN gives some details of the oxygen variant of the direct oxidation process, a method developed by Shell at Emeryville. The catalyst employed, silver on a carrier, has shown exceptional durability and, in the case of two commercial units, the original charge of catalyst has suffered no decline in activity over the year and a half's period of operation of the plant, another feature, not disclosed in detail, is the recirculatory system at the reactor which employs a hydro-carbon fraction as coolant.

Lending interest to the direct oxidation processes are current Union Carbide activities in this field at Fawley and the announcement by the I.C.I. that their Severnside works is to produce 35,000 tons a year of ethylene oxide by the direct route. And it will be interesting to see how soon the chlorhydrin process for propylene oxide will be challenged by a direct oxidation process.

* R. P. van Oosten, J.Inst.Pet, 1960, 46, 443, 347.

## Determining the Order of Chemical Reactions

WHEN the order of a chemical reaction is known, the factors influencing the speed of the reaction can usually be determined. Recently the U.S. National Bureau of Standards* have given details of a method which enables the order of a chemical reaction to be simply and rapidly calculated, with the aid of tables. The procedure can also be used to detect errors in initial times, initial concentration, stoichiometry; and in some cases it provides clues to the forms of complicated kinetic expressions.

The method of finding order is based on time-ratio tables calculated by the Bureau. For a given reaction, values were determined of the time $t_x$ required for the reaction to reach $x$-per cent completion. Successive overlapping ratio values ($t_{15}/t_5$, $t_{20}/t_{10}$, ...$t_{95}/t_{85}$) were then tabulated as a function of order to give the tables. Time-ratio values can be plotted as a function of order, as is done for several time-ratios. By using such a family of curves, fractional orders can be easily obtained.

To find the order of a chemical reaction by the Bureau's method, the degree of advancement of the reaction, i.e., the extent of the reaction completed, is obtained experimentally at specific intervals. This is done by measuring some physical or chemical property which is a known function of the concentration of a reactant or product. By plotting the different percentages of completion as a function of time, and drawing a smooth curve through these points, values of the times for various degrees of advancement can be obtained. Then, by comparing different ratios of these times to the time-ratio values on the Bureau table, the related order can be rapidy determined.

An induction period or initial period of rapid reaction, which may be too short to be recognized by other methods of kinetic analysis, will appear as an error in the initial time or concentration. To distinguish between an error in initial time and an induction period, a new degree of advancement can be calculated from an arbitrary point on the curve representing the concentration as a function of the time.

From the changes in apparent order for successive time ratios, approximate values for the parameters of a much more general type of differential rate equation can be obtained. This equation is applicable to data describing some reversible and simultaneous reactions as well as many catalytic and chain reactions and may prove useful in preliminary kinetic investigations of a chemical system.

* Technical Note 62, Department of Commerce, Office of Technical Services, Washington 25, D.C.

## Integrated Power and Processing from Low Cost Fuels

PILOT plants are now in operation in the U.S.S.R. and other countries for the production of city gas, tar and electric power in a continuous sequence of integrated plants. The process is based on the low-temperature carbonisation of solid fuel, the power-house boiler being fired with de-gassed fuel, i.e., semicoke; the mixture of steam and gas produced in the carboniser is separated into gas and tar in the subsequent condensation phase.

In an integrated power and processing plant, the heat value of the fuel is distributed approximately as follows:

| | |
|---|---|
| into the boiler ... ... ... | 50-55 per cent |
| into tar and city gas ... ... | 20-25 per cent |
| heat losses and plant consumption ... ... ... | 20-30 per cent |

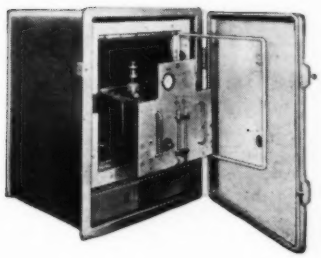
Assuming 20 per cent as the efficiency of the power plant (which, in these experimental installations, is of low pressure, temperature and capacity) the heat value distribution in the useful products of the plant is:

| | |
|---|---|
| Electric power ... ... ... | 10.4 per cent |
| Tar and petrol ... ... ... | 13.6 per cent |
| City gas (delivered to consumer) | 8 per cent |
| Total | 32 per cent |

With more modern steam power plant, an increasing proportion of the heat value of the raw fuel would be converted to electric power.

In the U.S.S.R., considerable development is being expended on the low-temperature carbonisation of low-grade fuels such as Esthonian shale, Moscow coal, peat, and Ukrainian brown coal, and pilot plants have been set up. Efficiency of carbonisation is 75-85 per cent depending on moisture content of raw fuel. An experimental plant

# Process Analyser



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fuelled by wood waste has already been operating for 7 years at 7,000 hr per year. The wood waste is gasified in a shaft in front of the grate of a 20 ton/hr steam boiler. By-products from the wood tar include calcium acetate, acetic acid, propionic acid, solvents, impregnating materials, drugs, herbicides, foundry binders and chemical compounds for the leather and rubber industries. A disadvantage of this process is the low heat value of the carbonisation gas, about 2,000 kcal/cu m and the impossibility of conveying it over long distances for public consumption.

The effective utilisation of tar is a decisive factor in the economy of the new integrated process. In the U.S.S.R. tar is being considered as a source of phenols and other aromatic compounds. It is estimated that the first cost of a combined power station and gas works will be 30-40 per cent higher than for a conventional steam power station, but considerably lower than for the construction of a separate power station and gas works.

## Distribution of Solids in a Piped Gas/Solid-Particles System

IN a solid-particle/gas stream flowing in a pipeline, the distribution of the solid particles across the stream may have significant effects on heat and mass transfer processes. With a view to throwing some light on the question, PROFESSOR S. L. SOO of the University of Illinois and J. A. REGALBUTO of Princeton University* have carried out experiments on the concentration distribution in a two-phase turbulent stream of high average velocity where the weight ratio of solid particles to gas was small, and hence the effect of gravity on the distribution was small.

The theoretical argument suggests that, due to their inertia, transport of solid particles across the velocity field of the turbulent stream by turbulent diffusion results in deceleration of the particles near the wall and acceleration of particles transported towards the high velocity core. In the core, the particles have lower mean velocity than the stream while near the wall the mean particle velocity is higher. This gives rise to a non-uniform distribution of solid particles in fully developed turbulent pipe flow.

The experiments were carried out with two sizes of solid particles, mean diameter 230 microns and 115 microns respectively, at loadings of 0.05, 0.10 and 0.15 lb of solid per lb of air, at average air velocities of 50, 75 and 100 ft per second. Concentration distribution was determined with an impact-disc particle counter system. Concentration distributions plotted against radius showed that the effect of gravity on the particles became negligible (1) as average stream velocity increased (average loading remaining constant) and (2) as the particle size decreased. The maximum concentration occurred, in some cases spectacularly, at the centre of the duct. The investigators conclude that in a two-phase turbulent stream of high average velocity, low loading, and with small particles, the concentration profile of solid particles in steady fully developed turbulence would approximate to the turbulent velocity profile, due to the similarity of the transport processes. For these conditions, they consider, their experiments confirm the validity of assuming a constant particle-concentration distribution.

* The Finance Act, 1960. A Summary for Engineers. Engineering Industries Association, October 1960.

## The Finance Act, 1960

A GUIDE through the complexities of the Finance Act, 1960, as it impinges on the affairs of the engineering industrialist, has been published recently by the Engineering Industries Association*. Although the Act is devoid of any legislation likely to affect directly the affairs of engineering firms (apart from the increase in the rate of Profits Tax from 10 per cent to 12½ per cent), "Summary for Engineers" sets out in simple terms the "intentions" of all sections of the Act likely to have even an indirect affect on engineering industries. Because of the intricacies of the Act, "all problems of precise interpretation must be left to the technicians". The sections covered are: Part I, Customs and Excise (Sections 1 to 14); Part II, Income Tax (Sections 15 to 43); Part III, Income Tax and Profits Tax (Penalties and Assessments); Part IV, Estate Duty (Sections 64 to 66); and Part V, Miscellaneous (Sections 67 to 79).

* The Finance Act, 1960. A Summary for Engineers. Engineering Industries Association, October 1960.

## DIARY

### Institution of Chemical Engineers

**January 24.** Meeting of the Institution at the Geological Society. Burlington House, London, W.1, at **5.30 p.m.** "A survey of chemical engineering education and practice," by PROFESSOR R. EDGEWORTH JOHNSTONE.

**February 2.** *Graduates and Students Section.* Meeting of the North Eastern Group of the Institution at the Head Wrightson Apprentice Lecture Theatre, Thornaby-on-Tees, at **6.45 p.m.** "Cooling with Air," by D. KAYE.

**January 24.** *North Western Branch.* Birkenhead Technical College, at **7.30 p.m.** "Experimentation and reactor design in process research and development," by W. WADDINGTON and W. D. BETTS.

**February 15.** The Geological Society. Burlington House, London, W.1, at **5.30 p.m.** Hinchley Memorial Lecture. "Science and the developing countries," by PROFESSOR P. M. S. BLACKETT. At Institution of Mechanical Engineers, 1a Birdcage Walk at **6.00 p.m.**

**February 21.** *North Western Branch.* Chemical Engineering Lecture Theatre, The Manchester College of Science and Technology, Jackson Street, Manchester, 1, at **6.30 p.m.** "Floating cap trays for absorption and distillation," by W. S. NORMAN (Member).

### Institution of Mechanical Engineers

**February 22.** Ordinary Meeting: Thomas Hawksley Lecture. "Controlled Fusion Reactions," by PROFESSOR P. M. S. BLACKETT.

### The Society of Instrument Technology

**January 31.** "Instrumentation Past, Present and Future," by L. S. YOXALL.

**February 6.** "The Universal Decision Element," by PROF. J. E. PARTON, Ph.D., M.I.E.E., and A. ROSE, M.Sc.

**February 28.** "Three Terminal Bridge Measurements and Automation," by DR. V. S. GRIFFITHS, Ph.D., B.Sc., F.R.I.C. These meetings will be held at Manson House, 26 Portland Place, W.1, at **6.30 p.m.**

### Institution of Engineering Designers

**February 2.** "Some Aspects of the History of Engineering Drawing Leading to Drawing Standardization," by P. J. BOOKER, A.M.I.E.D.

### The Institute of Refrigeration

**February 2.** "A New Conception in Cooling Tower Design," by CARL MUNTERS and LENNART LINDQVIST.

The Institution of Marine Engineers, Memorial Building, 76 Mark Lane, E.C.3, at **5.30 p.m.**

**March 2.** A symposium of papers on "Problems of Thermal Insulation and their Solution." (A joint meeting with the Low Temperature Group of the Physical Society, commencing at **2.0 p.m.**)

# COMPUTERS FOR THE CHEMICAL ENGINEER

THE Midlands Centre of the Graduates and Students Section of the Institution and the Graduates Section of the Birmingham University Chemical Engineering Society have arranged a joint one-day symposium on "Computers for the chemical engineer" which will be held on Tuesday, 28th March, 1961, at the Department of Chemical Engineering at the University, Birmingham 15.

Seven papers have been arranged dealing with methods, programming, plant management, research applications, and process control. Adequate time for discussion has been allowed during both the morning and afternoon sessions.

The titles of the papers to be given are as follows:—

"Computers and Programming" K. A. REDISH (Lecturer in Computing, University of Birmingham).

"Plant Management Applications" (D. G. THOMPSON, Esso Petroleum Co. Ltd., Fawley).

"Computers in Distillation Dynamics Research" J. E. RIJNSDORP, Koninklijke/Shell-Laboratorium, Amsterdam.

"The Need for More Process Control Computation" L. GALLON, British Petroleum Co., Ltd., Sunbury-on-Thames.

"Simple Computing Relays" R. J. REDDING and F. H. BODLEY, Evershed and Vignoles Ltd., London.

"Anatrol—An Analogue On-Line Process Control Computer" M. JAMES and W. T. LEE, De Havilland Aircraft Co., Ltd., Hatfield.

"The Argus Digital Computer in Process Control" W. F. M. PAYNE, Ferranti Ltd., London.

The opening paper by Mr. REDISH will give an outline of the types of problem for which analogue and digital computers are suitable. This is followed by descriptions of the various units and indicates the facilities that are required in a digital computer. The paper on plant management (D. G. THOMPSON) applications will be concerned with the use of digital computers in guiding Management for the control of investment, operations planning and cost control of a large oil refinery.

Mr. RIJNSDORP's paper will discuss the use of computers to study the dynamic behaviour of distillation columns. He will pay especial attention to the choice between digital and analogue techniques. In the case of the latter, which appear to be more suitable for control studies, the analogue circuit is divided into two parts; the pressure and flow circuit and the concentration circuit.

Mr. GALLON will survey current economic and technological trends in the process industries, and he will show how these trends provide opportunities for process control computation. In his paper he will review not only the possible uses of analogue and digital computers of varying degrees of complexity but also a number of techniques of automatic process optimization.

The multiple use of a computing device which can add, subtract, multiply and divide, is to be described by Messrs. REDDING and BODLEY. It is applied in the production of town's gas from the by-products of an oil refinery.

Messrs. JAMES and LEE will open their paper with a brief review of the current forms of process control and of the accuracy to be expected from measuring instruments. This is to be followed by a justification of the analogue process control computer, and a number of applications of the analogue control computer, "Anatrol," are to be given.

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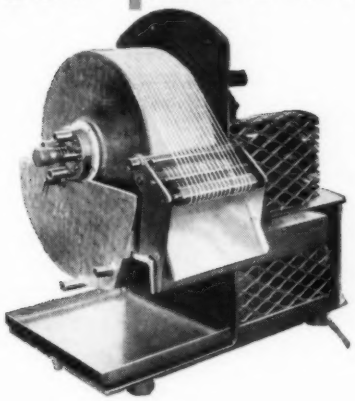

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DDT
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Gluten
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Rubber Reclaim
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Silica
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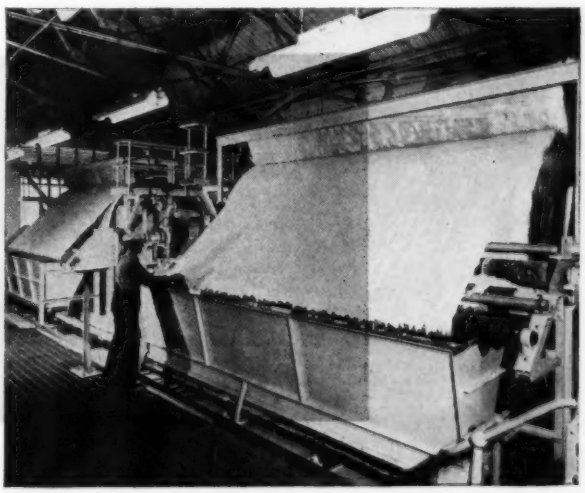

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|---|---|
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| 2 in. | 4-200 |
| 3 in. | 3-150 |
| 4 in. | 2-100 |
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| 8 in. | 1-40 |
| 10 in. | 1-30 |
| 12 in.* | 1-25 |

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# The Future of Ergonomics

ERGONOMICS may be described as the application of such varied sciences as anatomy, physiology and experimental psychology to the problem of fitting the job to the worker. This concept is not a new one and engineers in many fields have long made it one of the foundation stones of their designs. Whether a distinct calling of ergonomist will ever emerge is a subject for speculation; certainly there are people of standing who have practised ergonomics over many years without having been aware of it. Professionally speaking, ergonomics is at present a matter of specialists from different fields each making his own contribution to a given problem.

A number of examples of this have been given in recent times. In the steel industry heat stress is regarded as one of the possible stresses experienced in the working environment, and researchers in the field have evolved types of protective clothing which approach the desired goal of allowing men to work in great heat without suffering from heat stress. Another fruitful field is in the design of instruments. A great deal of work has been done on this topic over the past two decades, stimulated first by the need of aircraft pilots, and more recently of controllers of automated equipment. Surprising increases in efficiency of dial readings can often be obtained by relatively simple modifications; e.g., placing digits upright rather than radially oriented on circular scales. There may be mentioned the work of functional anthropometry, which aims at establishing norms and ranges of body dimensions. This work is of extreme importance to the development of protective clothing, the design of control stations and many other design problems. Some of these, naturally enough, are quite remote from chemical engineering, but are touched upon here because principles established initially in one context have often been applied successfully to problems arising in fields remote from the scene of the original application. The importance of data derived from anthropometry to the designers of cabs for lorries and cranes is by now well known: it is useful to know the proportion of a population of drivers which can reach foot-pedals comfortably when seated. Another finding useful to design revealed that the mere act of getting in and out of the driver's cab on short-haul work required an energy expenditure comparable to that of heavy physical work. Thus the improved designs reduced significantly energy expenditure. Whether parallel applications can find their counterparts on a wide scale in the chemical industry is perhaps uncertain, but many maintenance operations could certainly be alleviated through a similar approach.

It is possible that the chemical engineer, particularly when engaged on the design of plants, will make use of ergonomics as yet another consulting service, much as he makes use already of, say, civil engineers or instrument designers.

A modern trend which ought not to be overlooked is the tendency for the proportion of office workers to increase compared with the proportion engaged directly in some kind of production. In the engineering field in particular there has been a considerable expansion in office activities, particularly with those aspects concerned with the manufacture of capital equipment goods. Whether this expansion has been accompanied by a thorough study of the working environment affecting engineers, chemical, mechanical, civil and others, as well as that of those important links in the design chain, the draughtsmen, is open to doubt; new premises as a rule automatically mean improved working conditions, but it is questionable whether they contain features based upon the principle of fitting the environment to the worker.

It is possible that another fruitful field is that part of the engineer's job related to the acquisition of information and its retention. Here, naturally, other approaches are possible, and indeed necessary; one can, for example, apply the unit operations approach—so familiar to chemical engineers—and regard information as a chemical raw material to be collected and processed much in the same way as any other. But it seems fairly certain, now that the principles governing information transfer, storage, and use are beginning to be understood, that a place will be found for ergonomics among the techniques used for tackling this vast problem.

Invariably, any new approach encounters resistance to its widespread acceptance, and ergonomics is no exception. Aside from the understandable suspicion of the down-to-earthers that this may be yet another newfangled occupation for people detached from hard realities, there is the oft-voiced complaint that its wider usage is hindered by the lack of communication between people consciously working in ergonomics on the one hand, and industry as a whole on the other. As a consequence, despite the wide range of disciplines which the subject embraces, many a useful finding has failed to reach those to whom it would have been of benefit. To some extent this difficulty is being tackled by a body much mentioned in this column—Warren Spring Laboratory—which is now acting as a clearing house for information on human sciences.

How formal contact between industry and science, clients and consultants, one might say, can be established is an important question requiring an answer if ergonomics is to be useful. Two alternatives may be suggested—one may either teach already trained scientists about industry, or one may take trained engineers or managers and teach them some biology. Of the two, experience gained so far favours the latter approach.

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# EFFECT OF PARTICLE SIZE DISTRIBUTION IN FLUIDISATION

## PART I

## The Preparation and Analysis of Particles

Investigations on gas/solid fluidised beds are limited in accuracy and extent by the methods of preparing and analysing particle sizes and their distributions. Since the methods used in previous investigations have not invariably been reliable, some of the conclusions drawn are of doubtful validity. In this article methods of preparing particles in the sieve and sub-sieve ranges on glass ballotini and microspheroidal cracking catalysts are examined

by W. J. THOMAS, P. J. GREY* and S. B. WATKINS

THE process of fluidisation is established with a considerable literature of its own. The theoretical equations of KOZENY,[1] CARMAN,[2] BLAKE[3] and LEVA[4] are well known, and adequately surveyed in a recent book by LEVA.[5]

Despite the extensive literature, there is still a great deal to be understood about the mechanisms involved. There is a general failure to appreciate the significance of the effect of wide particle size ranges in the bed. Such beds, often fluidised with gas velocities many times the minimum fluidising velocity, present many problems not met with under idealised conditions. Stability and elutriation problems are not the least important.

Many analyses of the problem have over-simplified the condition to one of gas passing through an evenly mixed fluidised phase with an approach to piston flow. This condition generally occurs at the onset of fluidisation, but at higher gas rates two phases exist. One phase is homogeneous, and the other consists of gas passing rapidly through the bed in the form of bubbles. The bubble history is controlled by the tendency of the gas to separate from the fluidised phase, and solid material to rain through the individual bubbles, causing their collapse. The effect could be greatly influenced by the particle size distribution in the bed. Bubbles promote mixing and bed turbulence. It has been stated that with such turbulence an even distribution of particles within the bed should be expected. Residence time studies in many cases support this view. On the other hand, HALL & CRUMLEY[5] observed definite classification in a bed of Fischer-Tropsch catalyst. The average particle size for fresh catalyst decreased with increase in bed height. With used catalyst, it was found that although the most dense particles were found at the bottom of the bed, there was a minimum density a few inches below the top of the bed, and particles in the dilute phase were of considerably greater density than those in the top 18 in. of the dense phase. OSBERG and CHARLESWORTH[6] also established that a classification occurred within the bed.

Elutriation is a most important factor in determining the economics of fluidisation, and its extent influences the stability of bed operation. The elutriation of particles from fluidised beds has been studied by LEVA,[7] LEBOUX,[8] OSBERG and CHARLESWORTH,[6] GREGORY[9] and JOLLEY and STANTON.[10] As fluidising velocities above the terminal velocity of the smallest particle have been used with little carry-over, it is apparent that the bed can exert a retaining force on the particles. LEVA[7] carried out extensive research using sand, and later Fischer-Tropsch catalyst. The former he abandoned due to the adverse effect of electrostatic forces on the reproducibility of results. The effects of concentration and time were determined. The residual fines concentration in the bed was related to time by the equation $c = c_0\, e^{-k\theta}$, up to a certain critical concentration when the relationship breaks down. The so-called break-point when this occurs appears to be characteristic of the material being fluidised. $k$ is the elutriation velocity constant and is a function of driving force, gas velocity, fluid and particle properties. LEVA found $k \propto u^4$ regardless of the fines/coarse ratio and particle density. Only overall conditions were investigated, and in some cases LEVA obtained a 98 per cent material balance. OSBERG and CHARLESWORTH[6] evaluated $k$, and found the effect of particle diameter, bed depth and concentration. They did not obtain a sharp break-point when using Scotchlite beads.

There seems to be little known on the amount of freeboard to be allowed. LEVA suggests that there is no change in solids concentration in the gas 12 to 18 in. over the bed. However, industrial equipment allow freeboards of 18 ft[11] for a catalytic cracking regenerator, and 60 ft[12] in a Winkler gasifier.

Published results on fluidisation show considerable contradiction, lack of agreement, and, in general, the investigations have been limited to comparatively narrow particle size ranges. The present work was carried out to try to fill some of the gaps in our knowledge, concentrating on the examination of:

(a) a wide particle size range;
(b) classification in the bed;
(c) elutriation rates.

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* Work submitted for a University of London Ph.D.

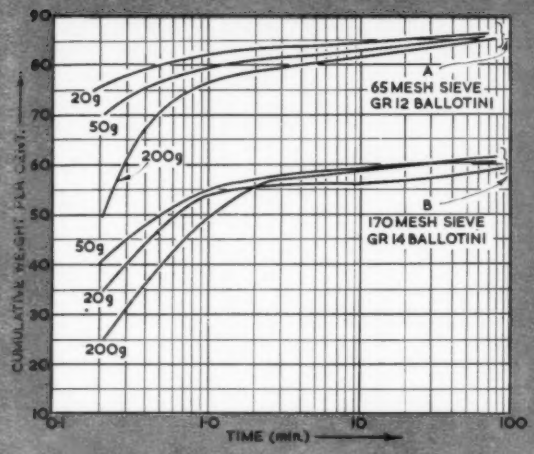


*Fig. 1. Sieve analysis with Sturtevant machine (1). Plot of cumulative weight per cent vs. log-time.*

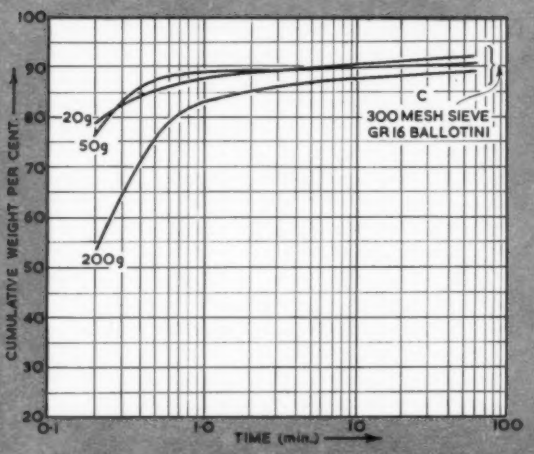


*Fig. 2. Sieve analysis with Sturtevant machine (2). Plot of cumulative weight per cent vs. log-time.*

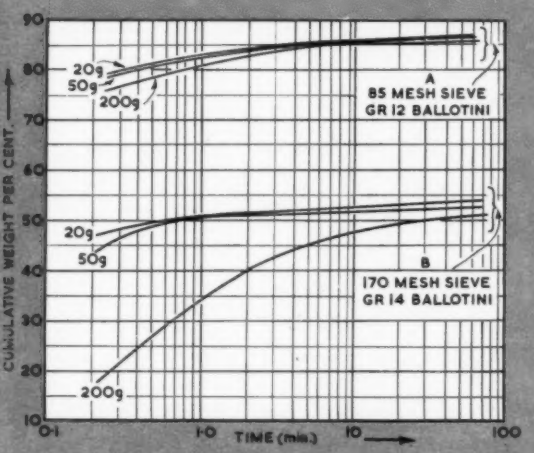


*Fig. 3. Sieve analysis with Ro-tap machine (1). Plot of cumulative weight per cent vs. log-time.*

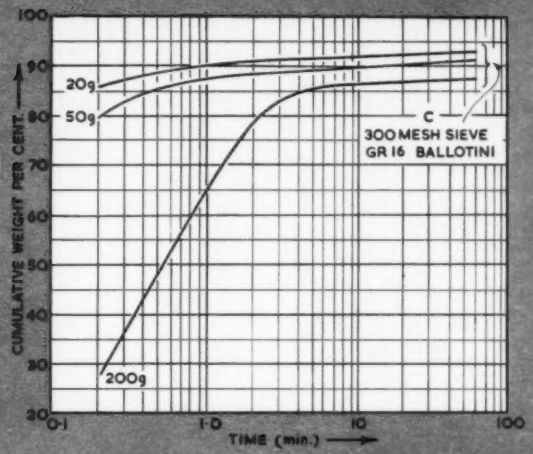


*Fig. 4. Sieve analysis with Ro-tap machine (2). Plot of cumulative weight per cent vs. log-time.*

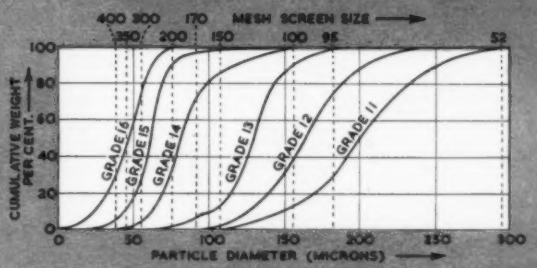


*Fig. 5. Sieve analysis of graded ballotini.*

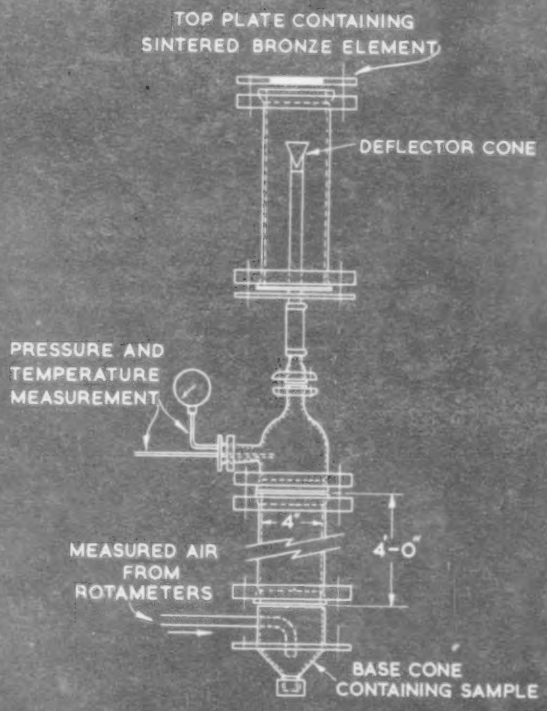


*Fig. 6. Elutriating column with reducing and separating sections.*

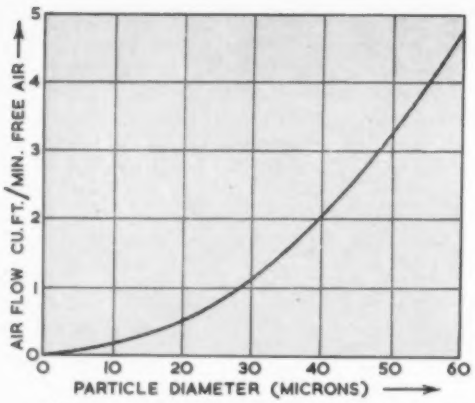


*Fig. 7. Air elutriator. Relation of elutriating air flow rate with particle size elutriated for glass ballotini; $\rho_s = 2.4$.*

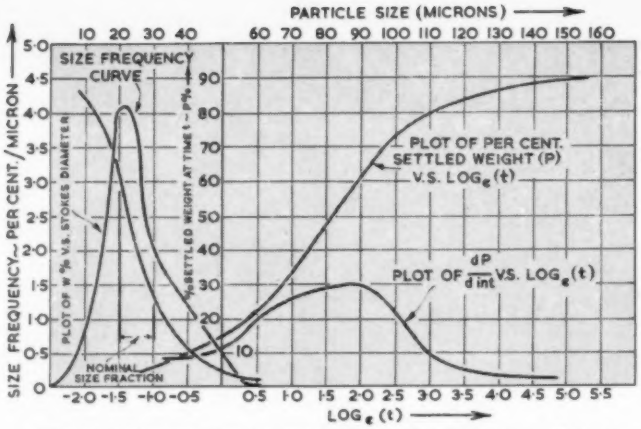
PARTICLE SIZE (MICRONS)
SIZE FREQUENCY CURVE
PLOT OF PER CENT. SETTLED WEIGHT (P) V.S. LOG$_e$(t)
PLOT OF $\frac{dP}{d\,int}$ V.S. LOG$_e$(t)
NOMINAL SIZE FRACTION
SIZE FREQUENCY ~ PER CENT./MICRON
LOG$_e$ (t)

*Fig. 8. Sedimentation balance. Size analysis No. 8. Ballotini Gr. 16, fraction elutriated 20-30 microns.*

The success of any such investigations is completely dependent on rapid, reliable methods for the preparation and analysis of particle sizes in samples. A special sampling technique was developed for studying particle classification within the bed. Accordingly, Part I of the paper deals with the preparation and analysis of particles, while Part II deals with fluidisation.

## The Preparation and Analysis of Particles

The diameters of particles in the fluidised bed must be so varied as to show the influence of the particle size distribution on classification within the bed, and on fines elutriation. Previous workers have used initial fines concentration differing widely from each other. For example, LEVA used 20 per cent of fines, whilst OSBERG used up to 5 per cent. The present research covers an initial fines concentration of from 2 to 20 per cent, so that the effect of concentration should be apparent.

It was decided to use glass ballotini and microspheroidal cracking catalyst. These materials were chosen to eliminate shape factors from the equations of flow, and the measurement of sub-sieve sizes. Both materials lend themselves to normal methods of grading. The ballotini was graded by sieving into five grades, followed by decantation and elutriation methods to obtain sub-sieve fractions.

### Sieves

The use of sieves for size separation is rapid and convenient. The sharpness of the cut depends on the shape of the particles, and to some extent on the shape of the openings in the mesh. The efficiency of sieving is influenced by the type of motion imparted to the screen. Vibratory motion is probably the best, followed by tapping.

Sieves were used in the present work for the preparation of beds containing particles in predetermined size ranges. They were also used for analysis of samples withdrawn from the bed, and of elutriated material. British Standard Test Sieves (BSTS), 8 in. for classification and 2 in. for analysis of small samples, were used. Sieving was mechanical, two machines being available for the purpose. The conditions under which these machines operate efficiently were unknown, and published information could not be found. The machines concerned were a Sturtevant vibrating sieve and a Ro-tap with hammer attachment. To cope with the extensive fluidisation programme, it was essential to obtain rapid, reliable results from the analysis of samples. The effect of load and time on the efficiency of sieving was determined using glass ballotini of various graded sizes. The total sieving time for each test was set at 60 min, the load being varied from 20 to 200 gm. Three different systems of material and mesh size were examined, and are given below.

(a) Grade 12 ballotini (nominal size range 145-190$\mu$) in conjunction with BSTS 85-mesh (sieve aperture 178$\mu$).

(b) Grade 14 ballotini (nominal size range 79-88$\mu$) in conjunction with BSTS 170 mesh (sieve aperture 89$\mu$).

(c) Grade 16 ballotini (nominal size range 10-60$\mu$) in conjunction with BSTS 300-mesh (sieve aperture 53$\mu$).

The effect of load on efficiency of sieving is quite marked, as is shown in Figs. 1 to 4. It is evident that a load of 50 gm sieved for 30 min provides a high efficiency of sieving. The two machines appear to be equally effective after 30 min. The vibrating sieve was used throughout the subsequent experiments with a loading of 50 gm over 30 min. A sieve analysis was carried out on all the grades of ballotini, and the results are given in Fig. 5. The nominal size ranges of these grades are given in Table I.

**TABLE I—Nominal Sizes Ranges of Graded Glass Ballotini**

| Grade | Microns |
|---|---|
| 11 | 190–295 |
| 12 | 145–190 |
| 13 | 88–112 |
| 14 | 79–88 |
| 15 | 60–79 |
| 16 | 60 |

The sieve analyses shown in Fig. 5 closely approach the nominal grading.

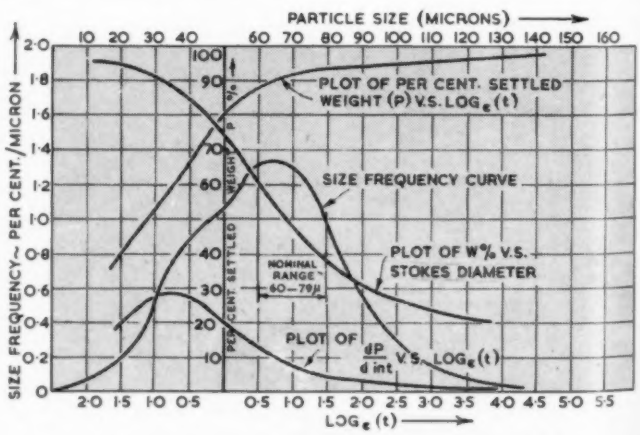
PARTICLE SIZE (MICRONS)
PLOT OF PER CENT. SETTLED WEIGHT (P) V.S. LOG$_e$(t)
SIZE FREQUENCY CURVE
PLOT OF W% V.S. STOKES DIAMETER
NOMINAL RANGE 60—79μ
PLOT OF $\frac{dP}{d\,int}$ V.S. LOG$_e$(t)
SIZE FREQUENCY ~ PER CENT./MICRON
LOG$_e$ (t)

*Fig. 10. Sedimentation balance. Size analysis No. 10. Ballotini Gr. 15.*

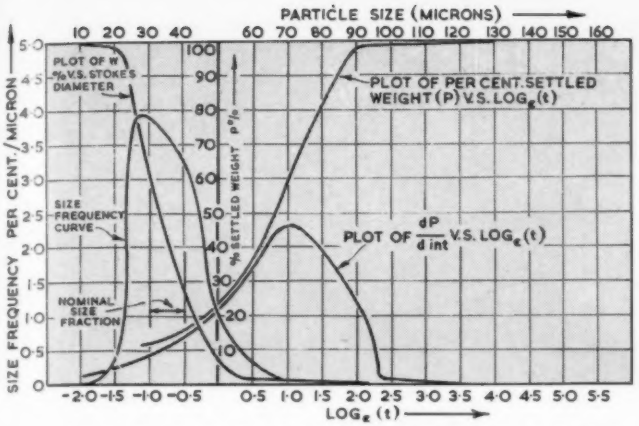
PARTICLE SIZE (MICRONS)
10 20 30 40 60 70 80 90 100 110 120 130 140 150 160
PLOT OF W %V.S. STOKES DIAMETER
PLOT OF PER CENT. SETTLED WEIGHT (P) V.S. LOG$_e$(t)
SIZE FREQUENCY CURVE
PLOT OF $\frac{dP}{d\,int}$ V.S. LOG$_e$(t)
NOMINAL SIZE FRACTION
SIZE FREQUENCY PER CENT./MICRON
% SETTLED WEIGHT P%
LOG$_e$ (t)

*Fig. 9. Sedimentation balance. Size analysis No. 9. Ballotini Gr. 16, fraction elutriated 30-40 microns.*

## Elutriation of Sub-sieve Sizes

The process of elutriation is well described in the literature. Generally, either air or water is used as the elutriating medium. The former has the advantage of dry classification and the ability to handle large quantities. Well-known elutriators using water are those of ANDREASON and ANDREWS. When elutriation is carried out in a tube, the velocity distribution pattern over the cross-section is such as to limit the sharpness of the cut. STAIRMAND[13] gives analyses of particle size of samples of material elutriated by various methods, and in all cases the sample had a considerable "tail" falling outside the nominal size range of the cut. In a bed being elutriated, particles may often be seen to ascend near the middle of the tube at the point of maximum velocity, to cross the tube near the top, and to descend near the wall where the velocity is low.

The three most important air elutriators are those of ROLLER,[14] GONELL[15] and HAULTAIN.[16] Tests on these elutriators compared with photo-extinction analyses are given by STAIRMAND. The Haultain Infrasizer is superior both in the sharpness of the cut and the speed of separation of the particles. However, it is a costly piece of equipment not generally available. It was decided to use the Gonell type of elutriator as being superior to the Roller type.

## The Gonell Elutriator

The elutriating column (4 ft × 4 in. dia), the top reducing section and the separating section are of Pyrex glass. The construction is shown in Fig. 6. A conical brass sample container is attached to the base of the column. The inlet air tube is bent downwards to direct the air on to the top of the powder sample. This method of air introduction ensures the material is adequately circulated and mixed, and prevents choking of the air inlet which would set up pulsations in the air flow. Air and elutriated material pass into the separating chamber, an inserted conical deflector on the entry pipe serving to knock the particles out of the air stream. Particles collect at the bottom of the separating chamber while air passes out through a sintered bronze disc. The elutriator was tested on glass ballotini.

Using the Stokes equation for falling spheres:

$$\frac{H}{t} = \frac{d_s^2\,(\rho_s - \rho_F)}{175^2\,\mu}.$$

For ballotini and air: $\rho_s = 2.4$ gm/cc. $\rho_F = 0.001266$ gm/cc. $\mu_{air} = 0.00018$ poise. $d_s$ = particle dia microns. $H$ = height cm. $t$ = min. $Q$ = flow rate free air ft$^3$/min. $u$ = air velocity ft/min.

It follows that $Q = 0.0903$. $u$ and $H/t = 0.4355$. $d^2_s =$ falling rate. The plot of particle size v. free air flow rate in the elutriator is given in Fig. 7. Fractions of ballotini classified by the air elutriator at the flow rates shown in Fig. 7 have been examined for particle size distribution using a Gallenkamp sedimentation balance. The results are shown in Figs. 8, 9 and 10. The fractions are seen to be close to the nominal size range, as was also shown to be attainable by STAIRMAND,[13] and as such they compare favourably with other methods of air elutriation. The distributions show definite "tails" outside the nominal size range of the fractions. These deviations from sharp cuts naturally limit the accuracy of any size analysis by elutriation, but not to an extent as to seriously affect the theoretical examination of the problems of fluidisation. The deviations are due to the occurrence of differential elutriation.

Similar results were obtained for microspheroidal cracking catalyst.

## Sedimentation

This method of size separation of particles in the sub-sieve range is based on Stokes law for the free settling of particles in fluids. It is well described by TAGGART,[17] who gives photomicrographs comparing fractions classified by decatantion sedimentation and air elutriation. The method was used on microspheroidal cracking catalyst in water, but proved to be no more effective than air elutriation, and has the additional problem of restricted sample size. The samples being wet, the tedious process of drying has to be carried out. However, the method proved quite important for the density classification of Grade 16 glass ballotini. During material density measurements, it was discovered that the densities of coarse grades of ballotini were reasonably constant at 2.4 gm/cc, but the Grade 16 or sub-sieve grade showed a marked drop in density to 1.6 to 1.7 gm/cc. A small part of the sample was even sufficiently buoyant to float on top of the alcohol used for density measurement. Grade 16 ballotini is important to the study inasmuch as it comprises the

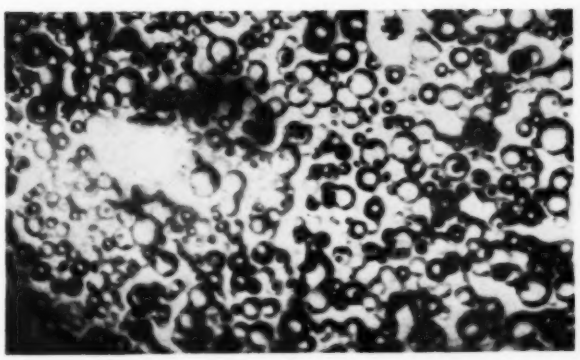

*Fig. 11. The photo-micrograph of Gr. 16 ballotini showing air inclusions and some glass bubbles.*

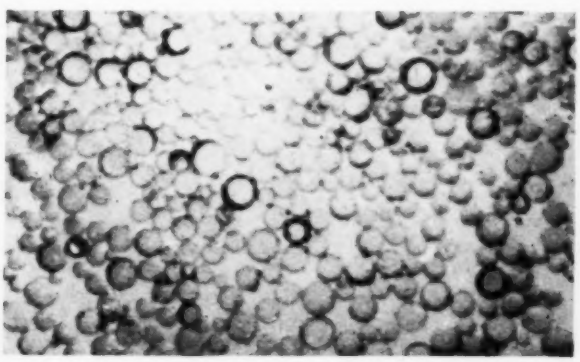

*Fig. 12. Ballotini of Fig. 11 after decantation sedimentation treatment.*

fines in a fluidised bed. The Grade 16 is not generally available in ballotini, and it had to be specially extracted from their plant by the suppliers. Photomicrographs of Grade 16 ballotini (Fig. 11) show a considerable part of the microspheres with air inclusions, and even glass bubbles are present. Many experiments with Scotchlite glass beads have been reported in the fluidisation literature, and it may well be that anomalous results and failure to correlate have been due to air inclusions and glass bubbles. It was considered vital to the success of the present study that all beads containing air and all glass bubbles be removed from the Grade 16 ballotini before use in fluidisation experiments. Ethyl bromide, density 2.18 gm/cc, was used in a decantation sedimentation treatment of the Grade 16 ballotini. Fig. 12 shows the ballotini of Fig. 11 treated in this way, and Fig. 13 shows the glass bubbles. It is seen that the inclusions of air have been reduced to a small amount, and that the glass bubbles have been eliminated. The treated ballotini had a density of 2.35 gm/cc compared with 2.4 gm/cc for the coarser material. The treated Grade 16 ballotini was used as the fines component in fluidisation experiments.

## Analysis in the Sub-sieve Range

Three methods were examined for the analysis of samples in the sub-sieve range.

(a) the Gallenkamp sedimentation balance;
(b) the photoextinction method;
(c) the microscope.

*(i) The Sedimentation Balance*

The apparatus is described in the Gallenkamp Catalogue No. 8078, and comprises a water-jacketed tube into which a suspension is introduced. The particles sediment on to the pan of a torsion balance, the settled weight being recorded optically. BOSTOCK[18] put forward the equation:

$$W = P - \frac{dP}{d(\log_e t)}$$

where $W$ = percentage of particles greater than a certain diameter;
$P$ = percentage settled weight at time $t$.

$P$ is plotted against $\log_e t$. If the curve is differentiated, and the resultant differential coefficients plotted on the same abscissa, then the difference between the ordinate of the two curves gives the percentage weight of particles greater than a certain size at any time within the range. The weight size distribution relationship can then be evaluated, and plotted as $W\%$ vs. Stokes diameter. This latter curve is finally differentiated to give the size frequency in per cent per micron against Stokes diameter; that is, the size distribution curve. This is shown in Fig. 14, Fig. 15 for microspheroidal catalyst and Figs. 16 and 17 for ballotini.

There is no universal dispersing agent for all powders, but it is essential that the suspension of the sample is well dispersed and homogeneous. The suspension can be examined microscopically to ensure good dispersion. Microspheroidal catalyst was dispersed readily in water with Teepol as dispersing agent. Glass ballotini could not be readily dispersed in this way. Various agents such as Dispersol T, Calgon, sodium citrate and organic solvents were tried. Finally excellent dispersion was obtained with isobutyl alcohol in water.

*(ii) Photo-extinction*

The purpose of the investigation of this method was to obtain a check on the analysis carried out with the sedimentation balance. The method is well described by ROSE,[19] and the particular apparatus used was the Evans electro-selenium photo-extinction apparatus. A parallel beam of light is projected through a dilute suspension of the sample



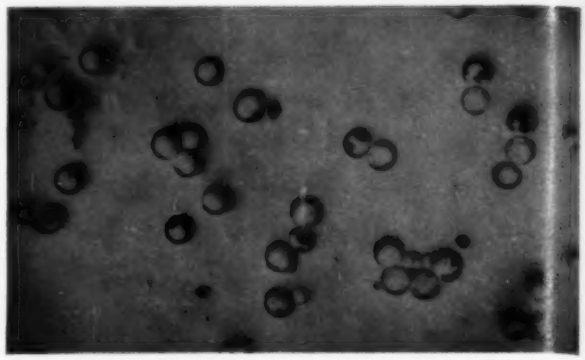

*Fig. 13. Glass bubbles removed from ballotini after decantation sedimentation.*

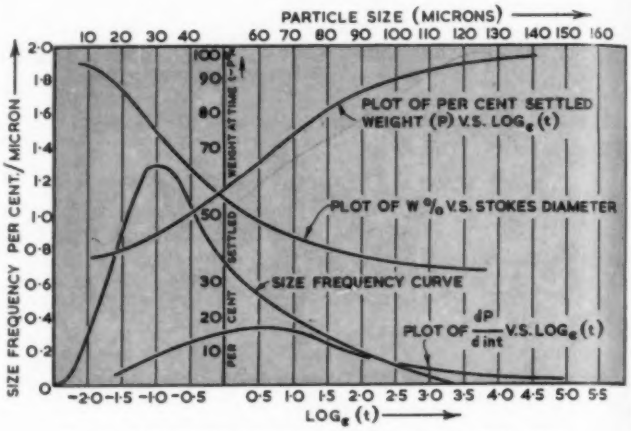
PARTICLE SIZE (MICRONS)
SIZE FREQUENCY PER CENT./MICRON
PLOT OF PER CENT SETTLED WEIGHT (P) V.S. LOG$_e$ (t)
PLOT OF W % V.S. STOKES DIAMETER
SIZE FREQUENCY CURVE
PLOT OF $\frac{dP}{d\,int}$ V.S. LOG$_e$ (t)
LOG$_e$ (t)

*Fig. 14. Sedimentation balance. Size analysis No. 1. Fresh microspheroidal cracking catalyst.*

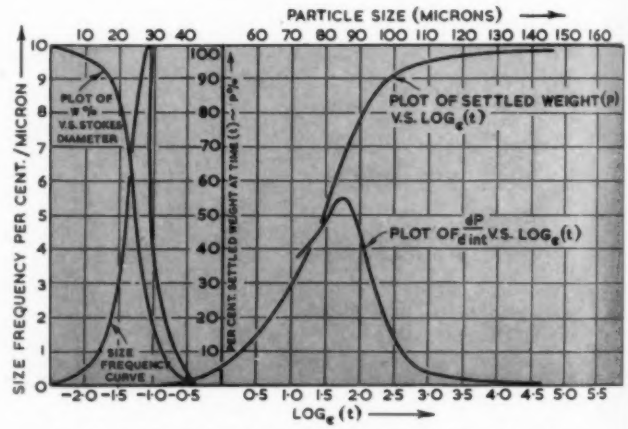
PARTICLE SIZE (MICRONS)
SIZE FREQUENCY PER CENT./MICRON
PLOT OF W % V.S. STOKES DIAMETER
PLOT OF SETTLED WEIGHT (P) V.S. LOG$_e$ (t)
PLOT OF $\frac{dP}{d\,int}$ V.S. LOG$_e$ (t)
SIZE FREQUENCY CURVE
LOG$_e$ (t)

*Fig. 15. Sedimentation balance. Size analysis No. 4. Microspheroidal cracking catalyst, 20-30 fraction.*

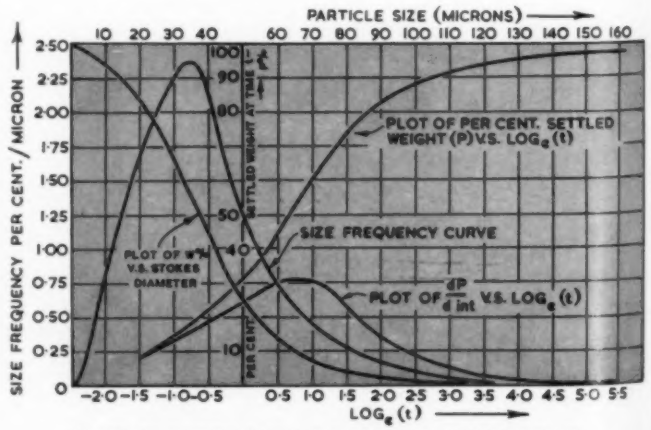
PARTICLE SIZE (MICRONS)
SIZE FREQUENCY PER CENT./MICRON
PLOT OF PER CENT. SETTLED WEIGHT (P) V.S. LOG$_e$ (t)
SIZE FREQUENCY CURVE
PLOT OF W % V.S. STOKES DIAMETER
PLOT OF $\frac{dP}{d\,int}$ V.S. LOG$_e$ (t)
LOG$_e$ (t)

*Fig. 16. Sedimentation balance. Size analysis No. 5. Ballotini Gr. 16*

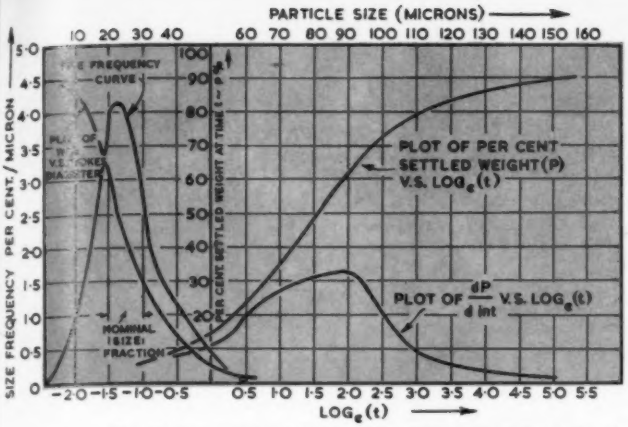
PARTICLE SIZE (MICRONS)
10 20 30 40 60 70 80 90 100 110 120 130 140 150 160
SIZE FREQUENCY CURVE
PLOT OF PER CENT SETTLED WEIGHT (P) V.S. LOG$_e$(t)
PLOT OF $\frac{dP}{d\,int}$ V.S. LOG$_e$(t)
NOMINAL SIZE FRACTION
SIZE FREQUENCY PER CENT./MICRON
PER CENT SETTLED WEIGHT AT TIME (t) = P
LOG$_e$(t)

*Fig. 17. Sedimentation balance. Size analysis No. 8. Ballotini Gr. 16, fraction elutriated 20-30 microns.*

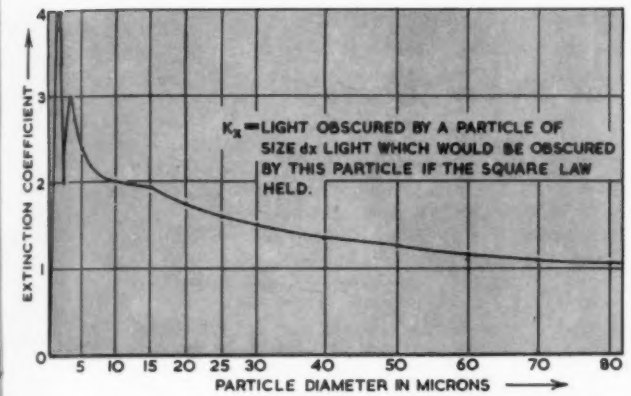
EXTINCTION COEFFICIENT
$K_x$ = LIGHT OBSCURED BY A PARTICLE OF SIZE dx LIGHT WHICH WOULD BE OBSCURED BY THIS PARTICLE IF THE SQUARE LAW HELD.
PARTICLE DIAMETER IN MICRONS

*Fig. 18. Photo-extinction size analysis. Plot of extinction coefficient vs. particle diameter.*

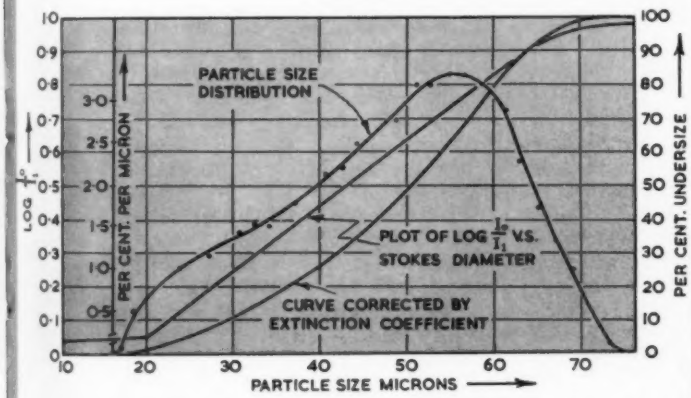


*Fig. 19. Photo-extinction size analysis No. 1. Sample of Gr. 16 ballotini. Dispersed in iso-butyl alcohol.*

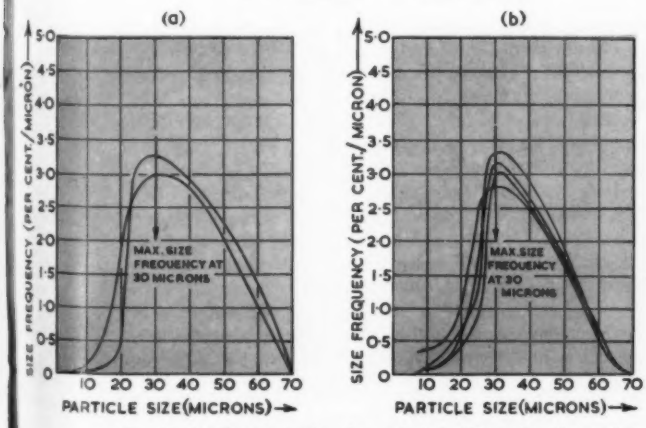


*Fig. 20. (a) Photo-extinction size analysis 3. Microspheroidal catalyst dispersed in iso-butyl alcohol.*
*(b) Photo-extinction size analysis 4. Microspheroidal catalyst dispersed in methylated spirits, repeat runs.*

on to a photoelectric cell. By using the Lambert-Beer law, it can be shown that:

$$\log_e \frac{I_0}{I_I} = k \,.\, c_I \sum_0^d K_x \,.\, n \,.d_x^2$$

where $I_0$ = intensity of illumination transmitted by clear fluid;
$I_I$ = intensity of illumination transmitted at a definite time after commencement of sedimentation;
$k$ = a constant depending on the shape and orientation of particles;
$c$ = concentration of the sample in the dispersion;
$n$ = number of particles;
$d_x$ = particle diameter;
$K_x$ = ratio of the light-observing power of a particle diameter $d_x$ to that for the same particle if the square law of geometric optics were applicable.

In the analysis, $\log_e I_0/I_I$ is plotted against $d$, where $d$ is the Stokes diameter of the largest particle which will reach the sampling zone from the top of the suspension in a measured time interval corresponding to $I_I$. This curve has then to be corrected for variations in $K_x$, the extinction coefficient, with particle size. This has been done for glass beads and plotted in Fig. 18. The weight percentage distribution curve is then found from:

$$\frac{d(W)}{d(d)} = \frac{\Delta \log_e I_I\, I_0}{\Delta d} \times d_s$$

where $d_s$ is the mean diameter in the interval $\triangle d$.

The results for Grade 16 ballotini are given in Fig. 19 and those for microspheroidal catalyst in Fig. 20.

The method proved to be simple and effective, and the results agree with those carried out on the sedimentation balance. In Figs. 14 and 20 it is seen that the maximum size frequency for microspheroidal catalyst occurs at 30 microns. The shape of the size distribution curves are also comparable, although the sedimentation analysis shows a slightly larger spread. The photo-extinction method has the advantage of allowing several samples to be examined at the same time.

*(iii) Microscope*

This is an absolute method for measurement of particle size, but as manual counts of particles involving a sample population of over 100 are tedious, then it can only be used as a check. The method was used and some photomicrographs are given in the present paper. The method is effective when used for regular shapes as in the present case. By dusting a slide with the material, checks of size frequency were made, and the method was also used for examining the efficiency of dispersion of powders prepared for sedimentation size analysis.

## Summary

The influence of fines in a fluidised bed on classification within, and elutriation from, the bed is important. Too little is known about the effects of particle size distribution on stability, pressure drop, and other important aspects. A study of these problems is not possible unless material intended for experimental work is carefully prepared in known size ranges. Again the accurate analysis of particle samples is essential to successful investigation of effects in fluidised beds.

Part I gives a careful assessment of preparation of glass ballotini and microspheroidal catalyst for use in experiments. The efficiency of sieves is considered, the Sturtevant vibrator and the Ro-tap machines being effective with limited loads over a definite time.

In the sub-sieve range several types of elutriator were

considered, a Gonell type being chosen for air elutriation of material both in the preparation and analysis of samples.

Decantation sedimentation, while generally a tedious method, was found to be valuable for density classification of Grade 16 ballotini. By this means, glass bubbles and glass with included air bubbles were removed from the material to be used in the subsequent fluidisation experiments. Their presence in the samples produce density variations and anomalous fluidisation results.

The sedimentation balance and the photo-extinction apparatus are equally effective in analysing samples for particle size and distribution. By their use, samples from the fluidisation experiments can be analysed to determine the extent of classification and elutriation of particles resulting from various flow conditions.


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# A Low-temperature Plant for Heavy Water

THE production of heavy water is complicated by a number of difficulties. It is present in ordinary water to the extent of 1 part in 7000, which means that very efficient means of separation must be used to obtain heavy water, from this source, of 99.8 per cent purity, the degree of purity required for heavy water used in nuclear reactors. The separation factors for various methods of heavy water production show considerable differences; for example, separation factors of ten are attainable with electrolysis, but for simple water rectification the figure is a mere 1.05.

When choosing a process it would seem that the first choice would be the method offering the highest separation factor, but the economy of the process is influenced by several other considerations. Thus, the power requirements for dissociation by exclusively electrolytic methods are relatively high and specific power inputs of 120-150 kWh/g of heavy water have been quoted.

A plant of recent design by Sulzer Bros., now in operation at Ems, in Switzerland, has a specific power input far less than this—no more than 2-3 kWh/g of heavy water. The design of this plant recognises that, for this particular process, the optimum conditions require the concerted use of several methods; whether these can all be applied depends upon linking the heavy-water plant with other manufacturing processes using hydrogen. Thus at Ems three methods are combined, electrolysis, rectification of hydrogen and deuterium and the rectification of ordinary and heavy water. At this plant the electrolysers of the ammonia synthesis plant are connected in cascade for heavy-water production. Somewhat more water is fed to each stage than is split into oxygen and hydrogen by the electric current. Excess water is removed from the gases by condensation and provides the feed to the next stage. In this way, an approximately seven-fold preliminary enrichment is obtained. The preconcentrate, containing about 0.1 per cent $D_2O$, is dissociated completely into oxygen and hydrogen in the final electrolyser. The hydrogen, of which some 400 $sm^3$ is stored in a gas holder before liquefaction is at about −250°C in the low-temperature plant.

At the low concentrations occurring at this stage the deuterium atoms are present only in combination with hydrogen atoms in the form of hydrogen deuteride. Since the equilibrium between hydrogen, hydrogen deuteride and deuterium is frozen at this low temperature, the best product that can be attained by rectification is 100 per cent hydrogen deuteride corresponding to 50 per cent deuterium. In the Ems process 60 per cent hydrogen deuteride is obtained by rectification, after which it is burnt in a simple oxyhydrogen burner. This produces heavy water of 30 per cent concentration which is distilled under vacuum at a temperature in the vicinity of 60°C to yield 99.8 per cent heavy water.

An advantage of this process is that its two rectifying columns can be operated independently and that the final column can be used for regenerating concentrated heavy-water waste.

## Low-temperature Plant Equipment

The column producing the 60 per cent concentrate of hydrogen deuteride is of the Kuhn type and consists of a series of vertical tubes connected in parallel in which the rectification process takes place. Liquefaction is produced by a combination of compression, heat exchange and expansion machinery. For example, hydrogen is compressed initially to about 14 atma in three-stage piston compressors. The hydrogen stream is cooled to liquefaction point by passing it through a series of heat exchangers, each followed by an expansion turbine. The final heat exchanger takes the form of an ordinary Joule-Thomson exchanger as used in air separation plants.

The purifying equipment is of considerable importance, since, without it, the expansion turbines might become choked with ice. The purification process, which is required for the first commissioning of the plant, consists of cooling the hydrogen with liquid air and passing it through activated carbon filters. A supply of pure hydrogen is stored in a dry gas holder when the plant is out of operation.

## Compressors

Both the circulating compressor of the refrigerating plant and the feed compressor are oil-free, so that no equipment is required for removing oil mist downstream of the compressors.

The most important units in the refrigeration plant are the expansion turbines, and the installation at Ems is the first hydrogen liquefaction plant to rely exclusively upon such machines for producing cold. As a rule the refrigeration required for hydrogen liquefaction is obtained by evaporating liquid nitrogen, which means that the hydrogen liquefier has to incorporate a complete liquid nitrogen plant.

The turbines are of the centripetal type, and a number of difficult problems have been overcome in their design. For example, the distance between the cold turbine wheel and the warm lubricating bearing is no more than 30 mm, and yet the temperature difference is in excess of 250°C, a difficult shaft-sealing problem and also to be overcome, since the shaft rotates at about 100,000 rpm.

66.048.37

# SOME RECENT VAPOUR LIQUID CONTACTING DEVICES

An account of three designs of vapour liquid contacting apparatus, each of which causes only a very low pressure loss. They are a rotary column, a helical packing and a special co-current distillation device. Operating characteristics are given for the rotary column and for the spiral packing, while for the co-current plate recommendations are given for its mechanical design

A NEW vapour liquid contacting device intended primarily for use in the distillation columns of new Soviet oil refining and petrochemicals plants is the subject of a paper in a recent Russian publication.[1] Some of the columns for the new plants are intended to fractionate up to 5000 tons of feed stock daily, their diameters will be 6-8 m and their heights 25-30 m, so that a great deal of metal will be used in their construction. The fact that some existing distillation devices do not allow maximum vapour velocities in excess of 0.5-0.6 m/sec was one reason for seeking a new design of plate.

In recent years, work has been done on the study of hydrodynamics and mass-exchange with co-current flow of gases and liquids currents in straight tubes. It has been shown that the mass-transfer coefficient increases with increase in the relative speeds of the phases, which depends on the speed of the gas current and the coefficient of friction on the gas-liquid boundary. For parallel flow at high speeds the magnitude of the coefficient of friction is 2-3 times greater than the maximum coefficient of friction in counterflow. The investigations cited provide a basis for the development of the new device as shown in Fig. 1. Each plate has two openings; in the larger there is a pipe for the flow of the two phases, while the second smaller opening carries the overflow device. This consists of a pipe with a cup attached at the lower end to form the hydraulic seal. When the plates are assembled, each plate is so mounted that its overflow is coaxial with the "contact" tube on the plate beneath.

When it enters the annular gap between the contact pipe and the overflow cup, the vapour stream contracts and passes through the gap at a high speed, withdrawing with it the liquid flowing from the cup; the pressure reduction at the constriction assists the egress of liquid and reduces the height of the hydraulic seal in the overflow pipe.

On leaving the annular gap the gas-liquid current impinges upon the bottom of the plate above, expands and moves downwards at reduced speed, a combination of events causing the drops of liquid to separate from the gas. The liquid flows through the overflow to the plate below, while the vapour passes into the "contact" tube of the plate above. A baffle mounted diametrically prevents the separated droplets from reaching the outer walls of the contact pipes, and is an additional aid to drop-separation in the space between plates.

At the Moscow Institute of Chemical Engineering the hydrodynamics of the new design of plate were investigated, the plates having tubes with a cross-section 3-20 per cent that of the column. The vapour velocities were 3-4 m/sec, based upon the free column cross-section and from 45 to 50 m/sec in the contact zone; the results showed that the hydraulic resistance $\triangle p_d$ of "dry" plates could be found from the equation

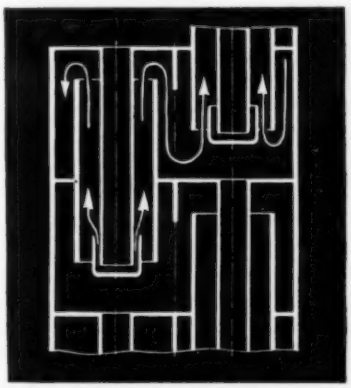

*Fig. 1. Diagram of vapour liquid contact device with co-current mass transfer tube.*

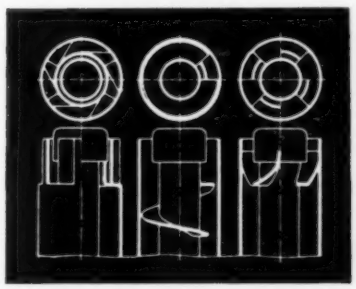

*Fig. 2. Swirl devices fitted inside the contact tube to promote greater mass transfer.*

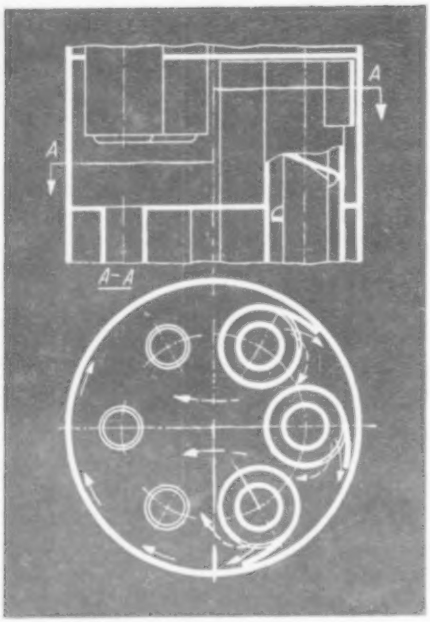


*Fig. 3. Diagram showing mechanical separation of the gas and liquid phases. Solid lines—movement of liquid; broken lines—movement of the vapour.*

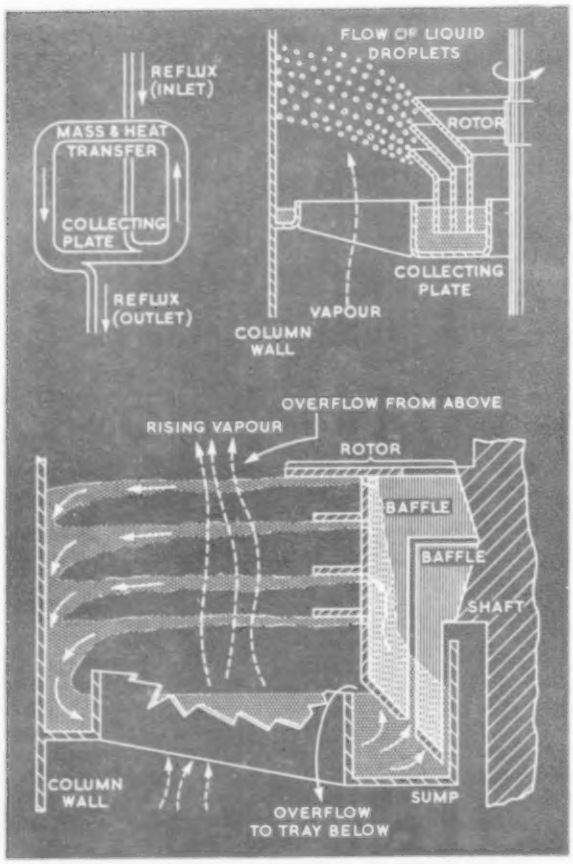


*Fig. 4. The operating principle of the Kirschbaum-Stora rotary mass transfer column and the Stora-Vulcan column. The rotating element in the latter differs in detail from that of the former.*

$$\Delta p_c = \varepsilon_d \frac{v_c^2 \rho_v}{2g} \; kg/m^2$$

where $\varepsilon_d$ = 1.8-2.0, the coefficient of local resistance per one contact plate;
$v_c$ = vapour velocity in the space between contact and overflow tubes, m/sec;
$g$ = acceleration of speed of gravity in m/sec$^2$;
$\rho_v$ = density of vapour in kg/m$^3$.

Visual observations on glass models revealed that at low air-flow rates the liquid in the gap between the cup and the contact pipe fell, and, consequently the plates were quite ineffective. When the velocity in the constriction was increased to 9-12 m/sec, the fall of the liquid ceased and it gradually filled the contact pipe in the form of an emulsion. This type of operation, the "emulsion" régime, is typified by intensive bubbling of gas through the suspended and heavily turbulent layer of liquid. It was observed that the greater the ratio of cross-sectional areas of plate and mid cross-sections of the "contact" tube respectively, the greater the range of gas-flow rates at which a suspension of the liquid occurs.

When the resistance of the gaseous medium exceeds the gravitational force on the liquid, the vapour stream ejects the liquid from the contact device, and the nature of the interaction of the currents changes abruptly. Depending on the content of gas in the liquid, flow régimes can be established analogous to those described in the literature for the flow of gas-liquid mixtures in straight tubes.

Experiments show that for the same vapour velocities based upon column cross-section, the overall resistance of plates of the new design is 5-6 times less than that of bubble-cap plates. However, since it is intended to operate columns fitted with these plates at superficial velocities considerably greater than those with bubble plates, the pressure-loss difference will in practice be less than this. For the majority of columns, however, it is the height of the hydraulic seal in the overflow device (on which depends the choice of interplate distance) that is the limiting factor, and not the hydraulic resistance itself. The new design of overflow device operates in more favourable conditions than the overflow pipes of grid-type and bubble plates, because a pure liquid, and not a foam-liquid mixture, flows over it. This makes it possible to decrease the hydraulic seal height by 1.5-2 times. The effect of injection also serves to reduce this height. Thus, despite the high vapour velocities, it is possible to have a plate-spacing of not more than 200-250 mm.

Further investigations led to improvement in this design. Vanes and baffles of various forms were fitted in the contact device (Fig. 2) to eddy the gas-liquid current. This feature provides much more contact surface and permits reduction in height of the contact device. This, however, increases the hydraulic resistance by 50-80 per cent. In addition, the outflow of the gas-liquid current from the contact pipe was modified (Fig. 3), so as to bring centrifugal force into play for phase separation with an increase (at atmospheric pressure) of the permissible vapour velocity in the main mass transfer section of 5/6 m/sec.

Given the densities of the two phases, and the vapour and liquor rates as obtained from a mass balance, a column using these contacting devices may be dimensioned from the following recommended figures for the various velocities which were obtained from extensive tests.

Vapour velocity in contact space, $v_c$ = 15-25 m/sec
Vapour velocity in the free cross-section of column where no special guide devices are employed, $v_f$ = 2-4 m/sec
Where such special devices are used, the velocity in the free cross-section of the column is, $v_f$ = 4-6 m/sec

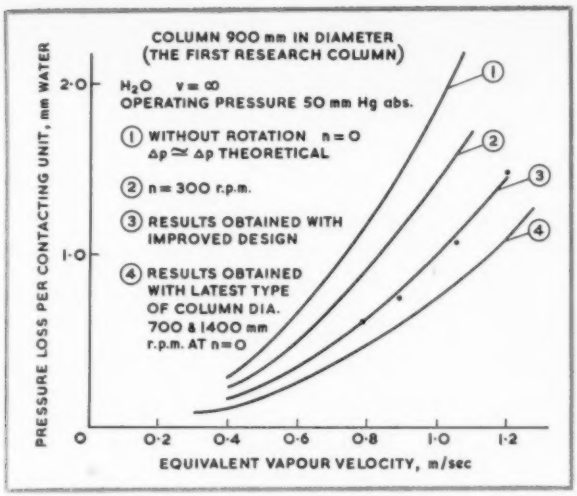
COLUMN 900 mm IN DIAMETER
(THE FIRST RESEARCH COLUMN)
H₂O v = ∞
OPERATING PRESSURE 50 mm Hg abs.
① WITHOUT ROTATION n = 0
Δp ≃ Δp THEORETICAL
② n = 300 r.p.m.
③ RESULTS OBTAINED WITH IMPROVED DESIGN
④ RESULTS OBTAINED WITH LATEST TYPE OF COLUMN DIA. 700 & 1400 mm r.p.m. AT n = 0
PRESSURE LOSS PER CONTACTING UNIT, mm WATER
2·0
1·0
0
0·2 0·4 0·6 0·8 1·0 1·2
EQUIVALENT VAPOUR VELOCITY, m/sec

*Fig. 5. Pressure loss characteristics for the Kirschbaum-Stora type of column.*

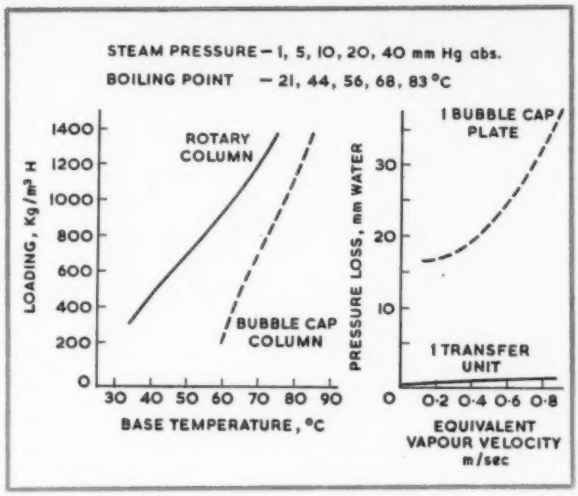


*Fig. 6. Performance characteristics of bubble-cap column compared with rotary column.*

The liquid flow at the seal-overflow should be, $v_{ov} = 0.10\text{-}0.15$ m/sec.

For high rates of mass transfer the length of the contact tube should be 200-250 mm. The distance between plates is then given by:

$$L = \tfrac{1}{2}(L_c + l_l + l_v)$$

where $l_l = G/\pi n d_c v_p$,

the distance from the lower end of the contact tube to the plate below, m, and

$$l_v = A_c/\pi n d_c$$

the distance between the upper end of the contact tube and the plate above, m.

The permissible velocity in the droplet separation part of the column $v_p$ may be found from the condition that the resistance forces, $T$, of the vapour are somewhat less than the gravity forces, $P$, on the liquid: i.e., $T = 0.8\text{-}0.9\,P$.

If the separated drops in the interplate space are considered to be spherical in shape, this condition may be formulated thus:

$$\frac{c\,\pi\,d^2\,\rho\,v_p^2}{8g} = 0.85\,\frac{\pi d^3}{6}\,(\rho_l - \rho_v)$$

$$\text{and } V_p = \left(1.1\,\frac{dg\,(\rho_l - \rho_v)}{c\rho_l}\right)^{\frac{1}{2}}$$

where $c$ is the resistance coefficient, a function of Reynolds number, and determinable by tests, and $\rho_l$ is the liquid density, kg/m³.

$d$ is the minimum allowable diameter of the drops in $m$.

Test data for an air-water system give values of $v_p = 6.5\text{-}7.5$ m/sec.

The length of the overflow pipe $L_{ov}$ can be found from:

$$L_{ov} = L_c + h_v + 0.03 \text{ m.}$$

(To form a reliable hydraulic seal, the overflow pipe should protrude by no more than 50 mm into the cup.)

The hydraulic resistance of the plates can then be calculated. Under operating conditions the height of the column of liquid in the overflow pipe must be 10-20 per cent less than the length of the overflow pipe: if this requirement is not met, the distance between the plates must be increased.

## Rotary Columns

Most of the established designs of plate column are not suitable for pressures below 10-20 mm Hg abs on account of their high pressure losses; and packed columns have the disadvantage that their effectiveness decreases with increasing vacuum, because the quantity of the reflux liquid present is too small to wet the packings adequately.

To overcome such shortcomings a number of designs have appeared including film columns, rotary columns without liquid circulation, and columns with evaporation and condensation occurring in a direction crosswise to the main movement of the phase. The Kirschbaum-Stora rotary column in particular represents an important contribution in this field and so far it has been supplied in diameters of 500, 700, 1000 and 1400 mm.

Invented independently by Kirschbaum whose work upon it has been reported elsewhere, and the firm Stora Kopparbergs Bergslags A.B. Falum, Sweden, this column has been brought to greater technical maturity by Muller-Schuss K.G., Weidenau. A similar design has appeared recently in the U.S.A.—the Stora-Vulcan column (Fig. 4). Perhaps the most noteworthy feature of these columns is that they replace conventional plates by units which consist of a system of collecting plates and rotors, the latter being attached to a shaft driven from outside the column. The action of the rotor is to discharge the liquid from the central collecting plate and to throw it against the column wall where it spreads and flows down into the outer channel of the collecting plate, from whence it returns to the central portion. Thus, in each unit, the liquid circulates continuously with its contact area formed in the path between the rotor blade tips and the walls of the column, an important feature being that each stage provides positive liquid mixing.

The vapour passes through the mass of liquid droplets, a feature which contributes to the very low pressure drop. Compared with bubble-cap columns, where the rising vapours, in passing through the bubble serrations, are accelerated nine- to thirteen-fold, in the case of the Kirschbaum-Stora column the vapour velocity is accelerated 3-4 times only. In addition, since there is no hydrostatic pressure drop through submergence of bubble caps, etc., this factor is absent from the total pressure drop.

## Recent Results

Recently results have been obtained at the Institute of Apparatus Design in Karlsruhe with a 900-mm rotary column of this type, while other results are available from the Bayer Manufacturing Co. for a 500-mm column. Results from the former are shown in Fig. 5. The loading in this diagram is expressed as equivalent vapour velocity in m/sec and corresponds to the "F" factor used in American literature and which is related to the equivalent velocity by

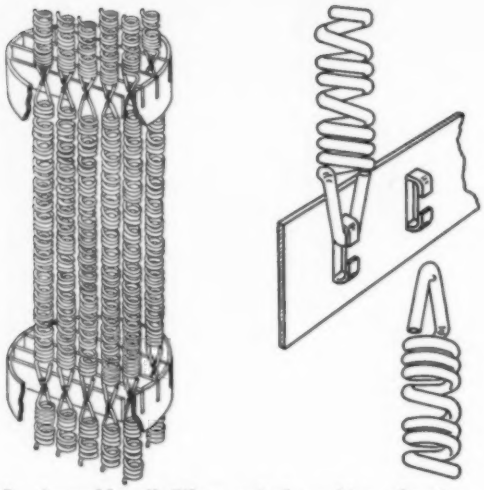

*Fig. 7. Assembly of Kloss spiral packing showing method of support and attachment by means of punched-out clips.*

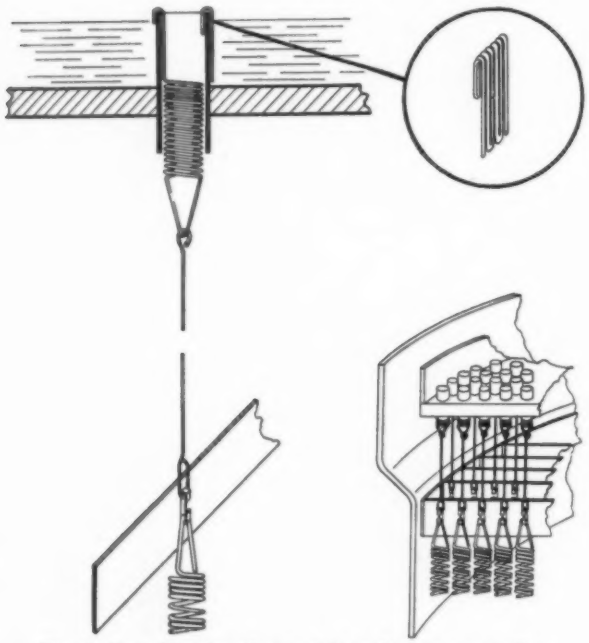

*Fig. 8. Detail of distributor. This relies upon capillary action for its effectiveness.*

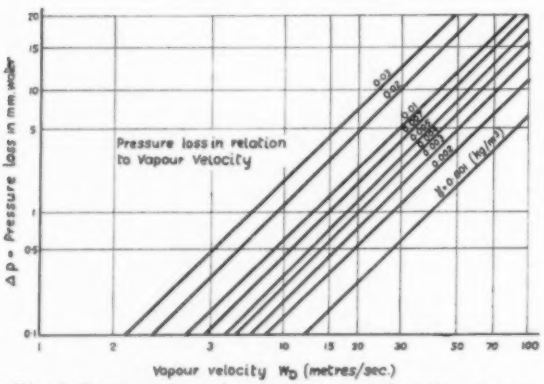


*Fig. 9. Pressure loss characteristics of the Kloss packing. (Pressure losses per m height of column.)*

$$w_L \text{ (m/sec)} \times 0.91 = \text{F-factor } \frac{\text{ft}}{\text{sec}} \frac{\text{lb}^3}{\text{ft}}$$

Available scanty mass transfer data indicate that a single actual stage of this device will produce a greater separation than a bubble-cap tray, and this is accompanied by a pressure loss almost independent of load. Curve four of Fig. 5 shows, compared with the first research column, a 50 per cent reduction in pressure loss for a normal operating load, the loss being less than 0.1 mm per plate. The favourable effect of this extremely low pressure loss upon still temperature on the one hand and the throughput on the other is shown in Fig. 6. This shows a comparison between a rotary column and a bubble cap column of equal diameters with ten actual plates. While in the bubble-cap column a still temperature less than 60°C is virtually unattainable, the corresponding temperature for the rotational column is less than 30°C for the condition of Fig. 6.

Two fields of application of this type of column are where (a) materials must be distilled over a narrow temperature range because, on the one hand, the base product cannot tolerate a high temperature and, on the other hand, because the head product, if there is too great a pressure fall in the working column, is likely to solidify (on account of the lower condensing temperature); and (b) inert gas treatment under conditions of low absolute pressure with pure-countercurrent flow, where a controllable time of retention is required at the individual stages. A typical example of this kind of gas treatment is deodorising.

For reasons of construction, the actual number of trays in such a column is limited to between 10 and 15. The total height is low, so that it is not difficult for columns to be connected together to give greater numbers of plates without additional heat loss or additional reflux pumping.

## Low Pressure Drop Packing

Of other widely used gas-liquid contacting equipment, random-packed columns will give satisfactory performance at absolute pressures in the range 760 down to 25 mm Hg, but at very high vacua they are not of much use. This is because, in order to avoid high pressure losses, the packings can be irrigated with only a small quantity of liquid corresponding to the low vapour throughput. As a result, channelling occurs, thereby reducing the mass transfer contact area and therefore the capacity of the column. If one attempts to compensate for the decreased capacity by increasing the contact area, i.e., by increasing the height of the column, then, in turn, one increases the pressure at the base of the column.

One solution to difficulties of this kind is to replace the random packings by helical springs made from solid wire strip—the main feature of the Kloss system.* The liquid fed to such a column flows filmwise over the entire surface of the spirals which are arranged with variable pitch. The effect of this feature is to cause the liquid to accelerate down the steeper sections of the spiral and to decelerate as the pitch decreases, the result of the change being to promote a strong mixing action.

As a rule the springs are arranged to fill completely the cross-section of the column in which they are fitted. The springs are arranged on a 20-mm equilateral triangular pitch, being 12 mm od and 620 mm in length. Each contact element is hung on to transverse strips which form a plate grid, the spirals being looped over punched-out clips, an arrangement which provides a positive transfer of liquid from one spring to that immediately beneath.

The individual grids are connected together by sectional plate supports to give a rigid assembly which permits easy removal from the column.

* Manufactured in Britain by The Coppee Co. (Great Britain) Ltd., Coppee House, 140 Piccadilly, London, W.1.

### Distribution of Liquid

For distillation purposes the distribution of reflux can be obtained by connecting each tube of the dephlegmator to each contact element, but this system is complicated and is limited to batch operation. For continuous operation, a special type of distributor is employed instead, details of which are given in Fig. 8. This consists of a plate into which is fitted a number of short tubes projecting 20 mm above and below the plate surface and on the same pitch as the springs. Each tube is connected to a spring by a wire bridge which, in turn, is connected to wire clamps fitted over the upper edge of the tube, which, by capillary action, cause the liquid to flow to the contacting elements. The operation of the column requires, for a given quantity of reflux, a definite level to be maintained on the tray; further regulation of the liquid distribution can be obtained by varying the number of clamps.

The columns do not differ outwardly from packed columns, except that at the distribution and feed plate levels, conical extensions are necessary for the diversion of the vapour round the distributors.

### Operating Characteristics

Each spring is capable of handling 0 to 1.1 l/hr of liquid. At rates greater than this the liquid tends to splash on to adjacent springs. The contact area provided is 180 $m^2/m^3$.

Vapour velocities are in the region 8-50 m/sec, but higher velocities can be tolerated provided the reflux can be accommodated by the contact elements and provided also that the pressure loss entailed does not exceed the permissible limits. The best fractionating effect is obtained, however, within the range 8-12 m/sec, although higher velocities do not reduce the separating effect seriously. So far little has been published on the mass-transfer performance, although, as a general rule, the number of theoretical plates per metre of column height is 3-8, according to the material distilled and the working conditions.

Pressure loss data for vapours of different density are given in Fig. 9. These figures emphasise suitability for low-pressure duties. The contact elements can be supplied in any of the usual metallic materials of construction.

REFERENCE
V. V. POPLAVSKY. *Khim. Mach.*, 1960, **5**, 4.

## Continuous Stirred Tank Reactor Calculations

WHERE great accuracy is not essential, the charts given below provide a useful means for calculating conversion in continuous stirred tank reactors, both single reactors and reactors in series. The charts have been produced by LIANG-TSENG FAN* of the Department of Chemical Engineering, Kansas State University, from a solution of the material balance equations for stirred tank reactors using Newton-Raphson's iterative technique. The basic balance equation, expressed in non-dimensional form, is:

$$\frac{R}{2}\gamma^n + \gamma - 1 = 0$$

where $\gamma$ is the ratio of the exit concentration to the inlet concentration of the reactant and

$$R = 2k\left(\frac{V}{F}\right)C_0^{n-1}$$

- $k$ being the specific reaction rate constant;
- $V$ the effective volume in litres of the reacting mixture;
- $F$ the volumetric rate of flow of reacting mixture, in litres a minute;
- $C_0$ the inlet concentration of the reactant in gram-moles per litre; and
- $n$ the order of the reaction.

In the case where a number of continuous stirred tank reactors are operated in series under steady-state conditions, the solution can be obtained by repeated use *of the appropriate chart.*

* *Industrial and Engineering Chemistry*, November 1960, 921.

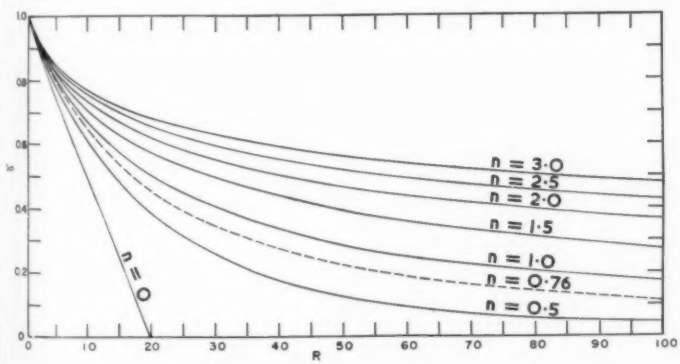


*Fig. 1. Determination of fraction unreacted for a given order of reaction.*

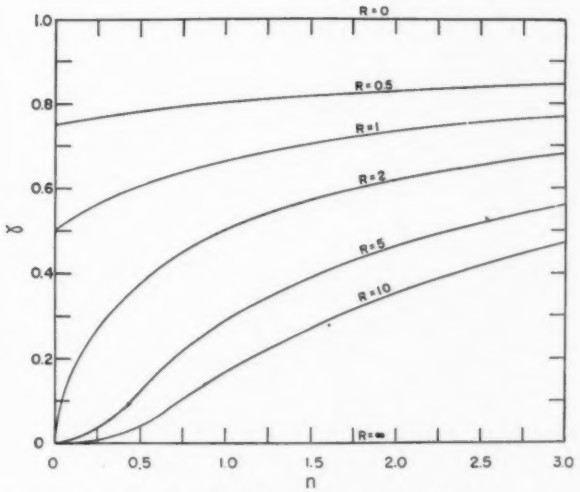


*Fig. 2. Determination of fraction unreacted when* R *is constant.*

541.123.2.012.6 : 66.048

# ISOBARIC VAPOUR-LIQUID EQUILIBRIA

This paper presents isobaric vapour-liquid equilibria data for the systems ethyl alcohol-ethyl benzene and ethyl alcohol-decane, and discusses the decrease in isobaric log activity coefficients in the dilute region of the concentration range for systems with wide boiling ranges

by S. R. M. ELLIS and M. J. SPURR

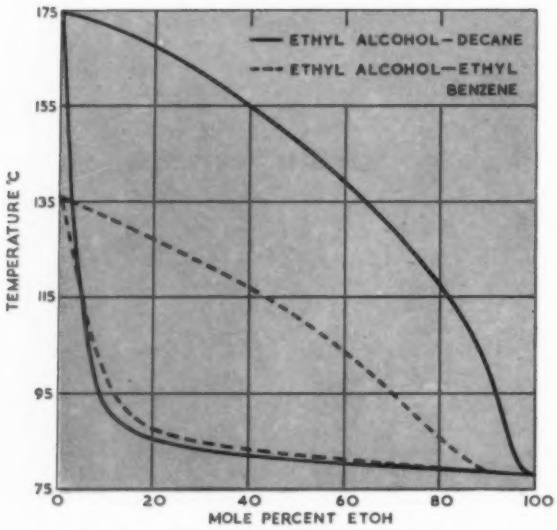


*Fig. 1. Experimental boiling point-composition curve for ethyl alcohol-ethyl benzene.*

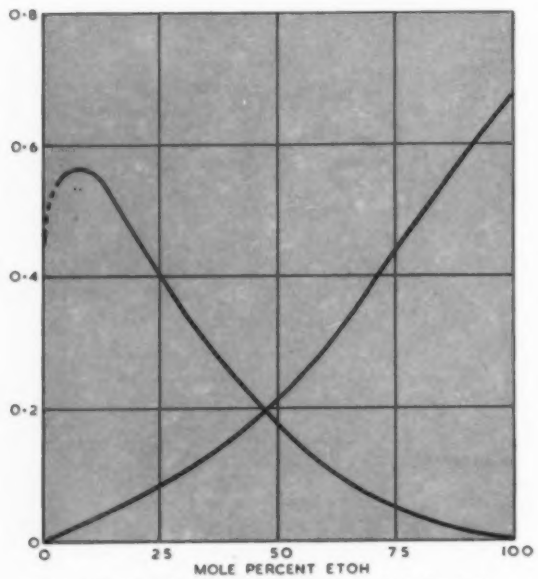


*Fig. 2. Log activity coefficients for ethyl alcohol-ethyl benzene.*

SOLUTIONS of the isothermal-isobaric Gibbs-Duhem equation are frequently used for the correlation of binary, isobaric equilibrium data. Their use, however, is somewhat limited, since boiling point differences between components of greater than 20-30°C can give rise to large discrepancies in the correlated log activity coefficients.

Experimental results are presented in this paper to illustrate these discrepancies, with particular reference to the rapid fall-off in the values of log activity coefficients corresponding to a sudden increase in the slope of the boiling point curve when the concentration of the more volatile component is small.

## Apparatus

The equilibrium stills used in this investigation have been previously described.[5, 6]

All components were carefully fractionated and purified such that there was close agreement with published results for refractive index, boiling point and density measurements.

TABLE I

| material | boiling point (°C) | | $N_D^{20}$ | |
|---|---|---|---|---|
| | experimental | literature | experimental | literature |
| ethyl alcohol | 78.3 | 78.3[11] | 1.3616 | 1.3614 [10] |
| ethyl benzene | 136.1 | 136.25[7] | 1.4958 | 1.4959[1] |
| decane | 174.0 | 174.0[8] | 1.4119 | 1.4120[13] |

Considerable care was taken with the operation of the equilibrium stills and in the measurement of pressures and boiling points.

Samples were analysed by accurate refractive index measurements.

## Results and Discussion

*Ethyl Alcohol-Ethyl Benzene*

The experimental results are given in Table II.

Activity coefficients were calculated from:

$$\gamma = \frac{\pi y}{Px}$$

where $\gamma$ = activity coefficient;
$\pi$ = total pressure;
$P$ = vapour pressure;

S. R. M. ELLIS is Professor of Chemical Engineering at Birmingham University and M. J. SPURR is a graduate research student in the same department.

$y$ = mole fraction of the more volatile constituent in the vapour phase; and
$x$ = mole fraction of the more volatile constituent in the liquid phase.

The correction factor, $z$, used for deviations from the ideal gas laws was evaluated from a nomograph by SCHEIBEL.[12]

Fig. 1 shows the experimental boiling point-composition curve for ethyl alcohol-ethyl benzene which increases rapidly when the liquid composition of ethyl alcohol decreases below about 10.0 mole per cent.

Also, in this dilute region, there is a corresponding fall-off in the log activity coefficient curve, making the extrapolation for an end-value very difficult, as illustrated by Fig. 2.

It would therefore appear that this decrease in the log activity coefficients for isobaric systems is associated with the slope of the boiling point curve, $\frac{dT}{dx}$, which may be large for non-ideal systems with wide boiling ranges.

IBL and DODGE[9] showed that for isobaric conditions the Gibbs-Duhem relation should be expressed as:

$$x_1 \frac{d \ln \gamma_1}{dx_1} + x_2 \frac{d \ln \gamma_2}{dx_1} = \frac{-H^M}{RT^2} \left(\frac{dT}{dx_1}\right)_p \quad \ldots . (1)$$

where $H^M$ = integral heat of mixing for a mole of the mixture; and
$T$ = boiling point of the mixture in °K.

HERRINGTON[8] and THIJSSEN[15] have introduced this temperature correction factor into the REDLICH and KISTER equation:

$$\int_0^1 \left[\ln \frac{\gamma_1}{\gamma_2} - \frac{H^M}{RT^2}\left(\frac{dT}{dx_1}\right)_p\right] dx_1 = 0 \quad \ldots . (2)$$

For convenience, IBL and DODGE refer to $\frac{H^M}{RT^2} \cdot \left(\frac{dT}{dx_1}\right)_p$ as $\overline{B}$.

Heat of mixing data by SMITH, BROWN and FOCK[14] for ethyl alcohol-toluene ($C_6H_5CH_3$) were used for the system ethyl alcohol-ethyl benzene ($C_6H_5.C_2H_5$).

The slopes of the boiling point curves were measured directly on a large-scale graph.

Table III gives the magnitude of the $\frac{\overline{B}}{2.303}$ values over the entire concentration range, and Fig. 3 shows the effect of this correction factor on the system ethyl alcohol-ethyl benzene.

For the log $\left(\frac{Z\gamma_E}{Z\gamma_{EB}}\right)$ vs. $x_E$ plot the ratio of the areas above and below the datum line is 0.792 with excess negative area. The introduction of the $\overline{B}$ factor into the REDLICH and KISTER equation, however, reduces the difference in areas, the ratio being 0.921.

*Ethyl Alcohol-Decane*

The experimental results are given in Table IV. From Fig. 4 it is observed that in the range 10→0 mole per cent ethyl alcohol there is a sudden fall-off in the values of the

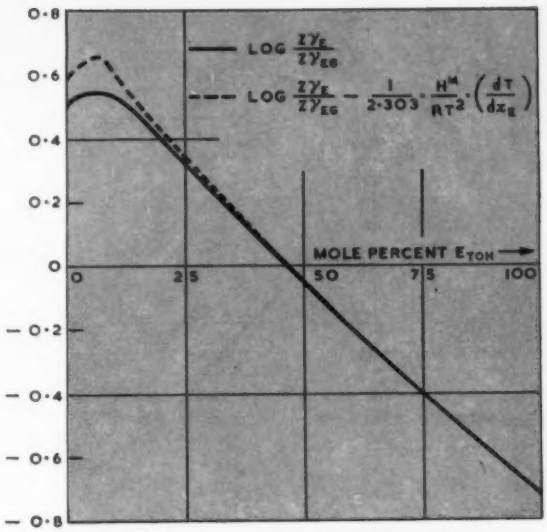


*Fig. 3. Effect of correction factor $\overline{B}/2.303$ upon log activity coefficient for the system ethyl alcohol-ethyl benzene.*

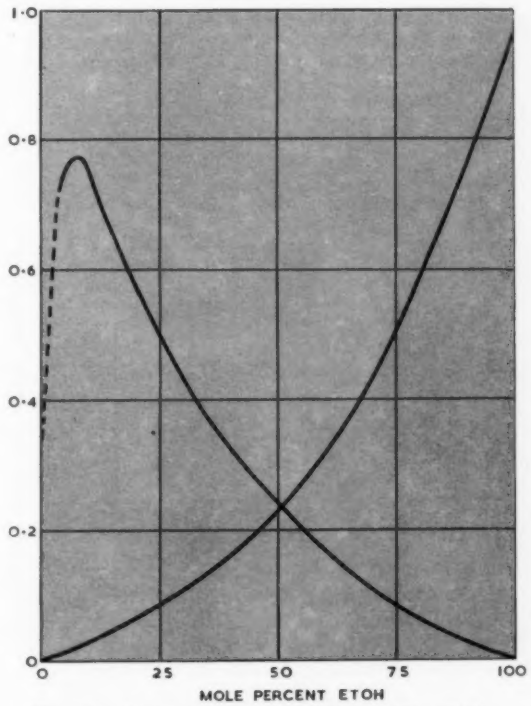


*Fig. 4. Log activity coefficients for ethyl alcohol-decane.*

TABLE II—Ethyl Alcohol—Ethyl Benzene 760 mm Hg

| t°C | mol. % ethyl alcohol | | ethyl alcohol | | | ethyl benzene | | |
|---|---|---|---|---|---|---|---|---|
| | liquid | vapour | $P_E$ | $Z$ | log $Z\gamma_E$ | $P_{EB}$ | $Z$ | log $Z\gamma_{EB}$ |
| 78.3 | 100.0 | | | | | | | |
| 79.1 | 82.0 | 90.3 | 1.026 | — | 0.027 | 0.1593 | 0.958 | 0.510 |
| 80.0 | 71.0 | 87.5 | 1.069 | — | 0.061 | 0.1655 | 0.958 | 0.397 |
| 80.8 | 61.6 | 86.3 | 1.094 | — | 0.106 | 0.1707 | 0.959 | 0.300 |
| 82.0 | 51.0 | 84.9 | 1.137 | — | 0.164 | 0.1785 | 0.960 | 0.218 |
| 85.0 | 26.0 | 81.5 | 1.274 | — | 0.390 | 0.1996 | 0.963 | 0.080 |
| 90.0 | 15.0 | 76.5 | 1.562 | 1.010 | 0.518 | 0.2394 | 0.967 | 0.047 |
| 95.0 | 10.8 | 71.3 | 1.804 | 1.017 | 0.570 | 0.2853 | 0.970 | 0.038 |
| 100.0 | 8.2 | 66.0 | 2.228 | 1.025 | 0.570 | — | — | — |
| 110.0 | 4.8 | 52.5 | 3.107 | 1.040 | 0.565 | — | — | — |
| 120.0 | 2.4 | 34.0 | 4.258 | 1.055 | 0.545 | — | — | — |
| 136.2 | 0 | | | | | | | |

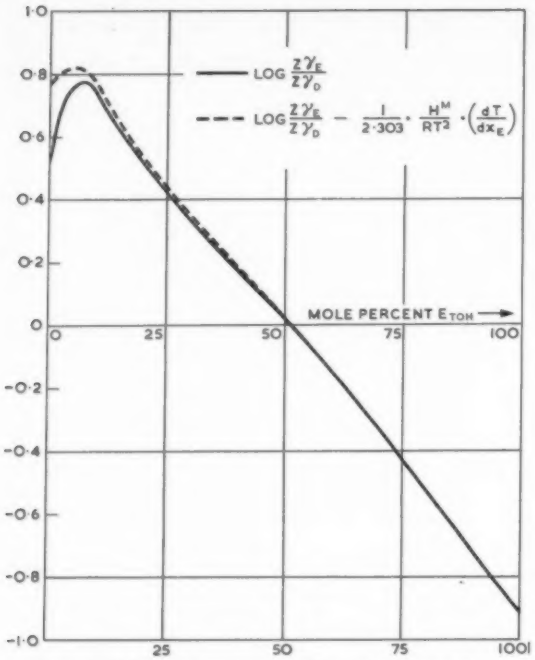
LOG $\frac{Z\gamma_E}{Z\gamma_D}$
LOG $\frac{Z\gamma_E}{Z\gamma_D} - \frac{1}{2{\cdot}303} \cdot \frac{H^M}{RT^2} \cdot \left(\frac{dT}{dx_E}\right)$
MOLE PERCENT $E_{TOH}$

*Fig. 5. Effect of correction factor $\overline{B}$ upon log activity coefficient for the system ethyl alcohol-decane.*

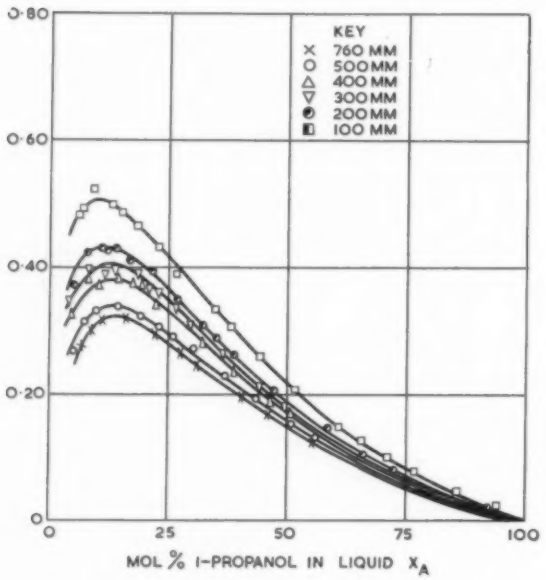


*Fig. 6. Activity coefficient curves for 1-propanol.*

**Symbols Used**

$t$ = temperature (°C);
$Z$ = SCHEIBEL correction factor for deviations from the ideal gas laws;
$\gamma$ = activity coefficient;
$\pi$ = total pressure;
$P$ = vapour pressure;
$y$ = mole fraction of the more volatile constituent in the vapour phase;
$x$ = mole fraction of the more volatile constituent in the liquid phase;
$T$ = absolute temperature (°K);
$H^M$ = integral heat of mixing for a mole of the mixture;
$R$ = universal gas constant; and
$\overline{B} = \frac{H^M}{RT^2}\left(\frac{dT}{dx}\right)_p$.

TABLE III

| $x_E$ | $\log \frac{Z\gamma_E}{Z\gamma_{EB}}$ | $\frac{\overline{B}}{2.303}$ | $\log \frac{Z\gamma_E}{Z\gamma_{EB}} - \frac{\overline{B}}{2.303}$ |
|---|---|---|---|
| 0.024 | +0.5360 | −0.075 | +0.6085 |
| 0.048 | +0.5490 | −0.1198 | +0.6688 |
| 0.082 | +0.5420 | −0.0668 | +0.6088 |
| 0.108 | +0.5320 | −0.0656 | +0.5976 |
| 0.150 | +0.4710 | −0.0264 | +0.4974 |
| 0.260 | +0·3100 | −0·0120 | +0·3220 |
| 0·510 | −0·0540 | −0·0032 | −0·0508 |
| 0·616 | −0·1940 | −0·0026 | −0·1914 |
| 0·710 | −0·3360 | −0·0017 | −0·3343 |
| 0·820 | −0·4830 | −0·0009 | −0·4821 |

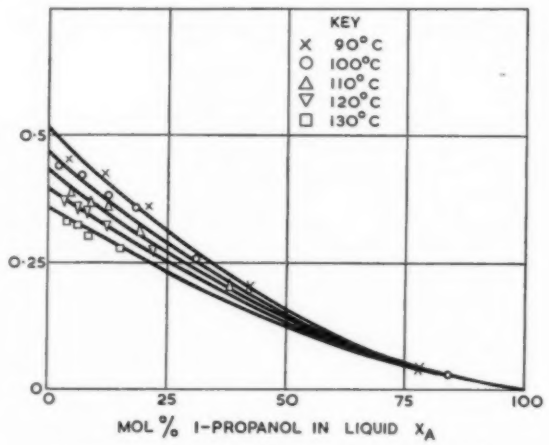


*Fig. 7. Isothermal activity coefficient curves for 1-propanol in nitrobenzene.*

log activity coefficients, corresponding to the sharp increase in the slope of the boiling point curve shown on Fig. 1.

In this case, the two effects are much more pronounced than for ethyl alcohol-ethyl benzene, and it is impossible to obtain an accurate, or even an approximate, end-value of the log activity coefficient for ethyl alcohol by extrapolation.

Fig. 5 shows the effect of the correction factor $\overline{B}$ on the REDLICH and KISTER equation.

The heat of mixing data for ethyl alcohol-decane ($C_{10}H_{22}$) was not available; thus data by SMITH, BROWN and FOCK[14] for ethyl alcohol-2,2,4-trimethylpentane ($C_8H_{18}$) were used.

For the log $\left(\frac{Z\gamma_E}{Z\gamma_D}\right)$ vs. $x_E$ plot, the ratio of the areas above and below the datum line is 0.980 with excess negative area. The modified REDLICH and KISTER equation slightly over-corrects, the ratio of areas being increased to 1.024 with excess positive area. However, it does tend to correct for the falling-off which occurs in the dilute region of the concentration range for ethyl alcohol.

CHAO[3] has also applied this correction factor $\overline{B}$ to isobaric systems with wide boiling ranges, and found that it may either give a significantly better correlation, over-correct,

TABLE IV—Ethyl Alcohol-Decane

| t°C | mol. % ethyl alcohol | | ethyl alcohol | | | decane | | |
|---|---|---|---|---|---|---|---|---|
| | liquid | vapour | $P_E$ | $Z$ | log $Z_{YE}$ | $P_D$ | $Z$ | log $Z_{YD}$ |
| 78.3 | 100.0 | | | | | | | |
| 79.1 | 91.0 | 97.7 | 1.026 | — | 0.019 | 0.0389 | 0.90 | 0.770 |
| 80.0 | 79.0 | 96.5 | 1.069 | — | 0.057 | 0.0401 | 0.90 | 0.572 |
| 80.8 | 62.0 | 96.2 | 1.094 | — | 0.150 | 0.0416 | 0.90 | 0.330 |
| 82.0 | 37.0 | 95.8 | 1.137 | — | 0.347 | 0.0439 | 0.90 | 0.130 |
| 85.0 | 20.0 | 94.8 | 1.274 | — | 0.590 | 0.0502 | 0.91 | 0.069 |
| 90.0 | 11.8 | 93.5 | 1.562 | 1.010 | 0.710 | 0.0624 | 0.91 | 0.027 |
| 95.0 | 8.6 | 92.0 | 1.804 | 1.017 | 0.780 | 0.0770 | 0.92 | 0.018 |
| 100.0 | 7.0 | 90.0 | 2.228 | 1.025 | 0.770 | — | — | — |
| 110.0 | 5.0 | 84.6 | 3.107 | 1.040 | 0.750 | — | — | — |
| 120.0 | 3.7 | 77.6 | 4.258 | 1.055 | 0.716 | — | — | — |
| 140.0 | 2.2 | 58.0 | 7.148 | 1.090 | 0.600 | — | — | — |
| 150.0 | 1.8 | 46.0 | 9.700 | 1.117 | 0.470 | — | — | — |
| 160.0 | 1.2 | 32.5 | 11.730 | 1.145 | 0.420 | — | — | — |
| 174.0 | 0 | | | | | | | |

TABLE V—Ethyl Alcohol-Decane

| $x_E$ | $\log \frac{Z_{YE}}{Z_{YD}}$ | $\frac{\overline{B}}{2.303}$ | $\log \frac{Z_{YE}}{Z_{YD}} - \frac{\overline{B}}{2.303}$ |
|---|---|---|---|
| 0.037 | 0.7090 | −0.0965 | 0.8055 |
| 0.050 | 0.7390 | −0.0804 | 0.8194 |
| 0.070 | 0.7540 | −0.0593 | 0.8133 |
| 0.086 | 0.7620 | −0.0347 | 0.7967 |
| 0.118 | 0.6830 | −0.0186 | 0.7016 |
| 0.200 | 0.5210 | −0.0088 | 0.5298 |
| 0.370 | 0.2170 | −0.0021 | 0.2191 |
| 0.620 | −0.1800 | −0.0011 | −0.1789 |
| 0.790 | −0.5150 | −0.0008 | −0·5142 |
| 0.910 | −0.7510 | −0.0004 | −0.7506 |

or give only a very slight improvement in the correlation. The applicability of the modified REDLICH and KISTER equation is also shown to vary considerably for the same system at different pressures.

Fig. 5, however, does illustrate how this correction factor can offset a sudden fall-off in the $\log\left(\frac{Z_{YE}}{Z_{YD}}\right)$ curve at low concentrations where there is a correspondingly rapid increase in the slope of the boiling point curve.

It is possible that this falling-off effect encountered with log activity coefficients at low concentrations of the more volatile component may be due to a decrease in the relative accuracy of the experimental data. For this reason CHAO did not use experimental data in which the composition of either component was below 5 mole per cent.

With these two systems there is a very rapid increase in the relative volatility for the range 10→0 mole per cent; consequently, there is a possibility that flash vaporisation may occur in the equilibrium still giving rise to non-equilibrium conditions. Also, the method for analysis of samples is least accurate for this region of the concentration range.

However, confirmation that this effect in wide-boiling, isobaric systems is not necessarily due to experimental inaccuracies can be obtained by cross-plotting the log activity coefficients for constant temperature at different pressures.

Figs. 6 and 7 illustrate this method for iso-propanol-nitrobenzene with data by FROOME.[6]

The isobaric log activity coefficients for iso-propanol in Fig. 6 show a tendency to fall-off rapidly at all pressures in the region 10→0 mole per cent iso-propanol.

Fig. 7 shows how cross-plotting the results on an isothermal basis eliminates the tendency of the isobaric log activity coefficients to rapidly decrease at low concentrations of iso-propanol.

## Final Discussion and Conclusions

The fall-off in log activity coefficients encountered with these isobaric systems at low concentrations for the more volatile component can be considerably reduced, or possibly eliminated, by the corrected REDLICH and KISTER equation, although, as CHAO has shown, it may over-correct or give only a very slight improvement.

The practical limitations of this temperature correction factor are governed by the availability of the appropriate heat of mixing data and the accuracy to which the slopes of the boiling point curve can be determined.

This work emphasises that for systems with wide boiling ranges there is a need for accuracy in the determination and measurement of the slope of the boiling point curve and for measuring experimentally the heat of mixing for the actual system being studied.


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## IN OUR NEXT ISSUE

It is intended to include the following articles in the March, 1961, issue of *British Chemical Engineering*:

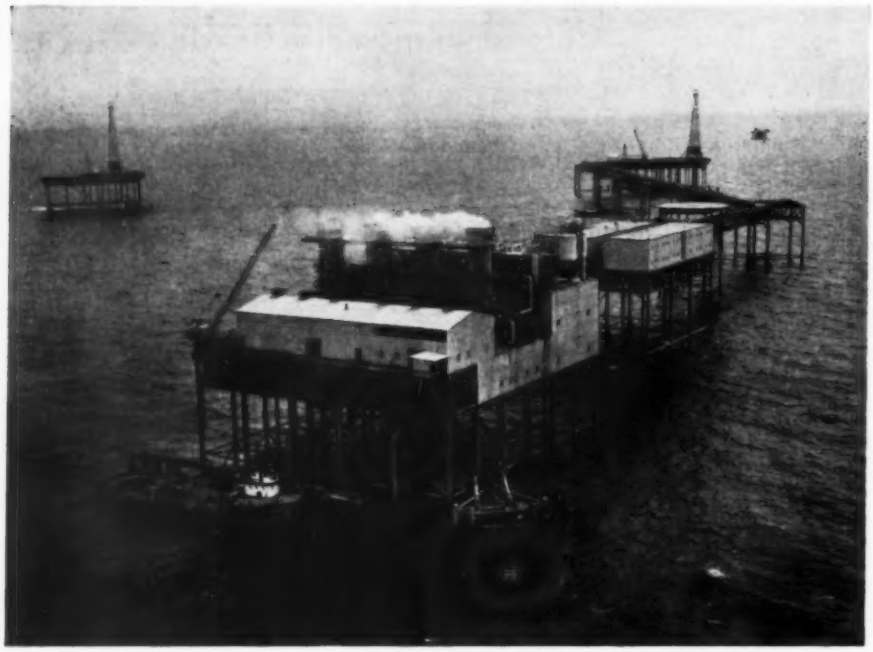
**Graphical Design of Bubble-cap Trays**
by L. S. GUOKHEGYI and J. J. CZERMANN

**Chemical Recovery in Swedish Sulphite Pulp Process**

**The Corrosion Resistance of Some Sprayed Metallic Coatings**
by T. K. ROSS and E. L. SMITH

**The Effect of Particle Size Distribution in Fluidisation—Part II**
by W. J. THOMAS, P. J. GREY and S. B. WATKINS

**A Survey of Distillation Equipment Costs**
by M. STOGENS



*General view of the world's first off-shore sulphur mine as it is today.*

# TRANSPORTATION OF SULPHUR FROM WORLD'S FIRST OFF-SHORE MINE

PROBABLY the greatest problem facing the engineers of the Freeport Sulphur Co., New York, U.S.A., when planning the world's first off-shore sulphur mine in the Gulf of Mexico, which has recently been placed in commercial production, was that of transporting the molten sulphur from the island of steel, seven miles from the Louisiana coastline, to the mainland. Normal surface methods of transportation were complicated by weather conditions at the exposed location and the use of conventional barges and tugs would have been risky. The solution was found in the use of a seven-mile-long heated pipeline, buried in the floor of the Gulf, through which the 300°F sulphur is pumped to a mainland base. Thence it flows into "thermos bottle" barges and is towed 25 miles to the Freeport Co.'s storage and shipping facilities at Port Sulphur on the Mississippi River. The pipeline, an innovation in sulphur industry history, is designed to handle 4500 tons of molten sulphur per day.

Buried about 5 ft below the bottom of the Gulf of Mexico, the pipeline consists of three concentric pipes. These comprise a 14-in. O.D. protective casing, a 7⅛-in. O.D. hot water jacket line and a 6-in. O.D. sulphur line. Liquid sulphur is pumped through the 6-in. pipe, while hot water is pumped through the annulus between the 6-in. and 7⅛-in. pipes. The space between the 7⅛-in. pipe and the 14-in. pipe is filled with a 2½ in. thickness of thermal insulation and a small air gap.

To enable the inner lines to be pushed or pulled through the seven-mile-long 14-in. casing and to permit virtually frictionless differential movement between the two sets of lines, the weight of the 6-in. and 7⅛-in. pipes is carried on 1½-in. O.D. ball-bearing rollers spaced at 19 ft 6 in. centres along the line.

The relative intake and exit temperatures of the sulphur and water have been fixed having regard to the excessive viscosity of sulphur at higher temperatures and also to the freezing temperature of sulphur, which is 240°F. Both sulphur and water are, therefore, taken into the pipeline transportation system at about 320°F, while the exit temperature at the shore end is about 280°F. An appreciable amount of heat is lost over the seven-mile journey, despite the high efficiency of the insulation and air gap. It is calculated that, with an average ambient soil temperature of 75°F, the 14-in. casing will reach about 110°F and that the heat loss to the wet soil is 90 Btu/linear ft/hr, or about 75 million Btu per day.

To maintain the maximum capacity of the pipeline of 4500 long tons of sulphur per day, or 470 gpm, a pumping pressure of about 900 psig is required at the mine end. At this rate of flow, only 15 gpm of 325°F water is required to assist in overcoming the heat loss. At a flow of 2500 tons per day, or 250 gpm, about 25 gpm of water is needed, while for starting up and at extremely low sulphur rates the water rate may reach 100 gpm. This requires a water pressure on the plant end of 1400 psig.

At the shore end, the sulphur is delivered into barges for further transportation to Port Sulphur, while the water from the 7⅛-in. line is cooled from 280°F to atmospheric temperature and returned through a 4½-in. O.D. line to the mine for reheating and reuse through the pipeline. This return line is strapped to the outside of the 14-in. casing of the delivery line, as also is a 6⅝-in. line which not only brings a limited

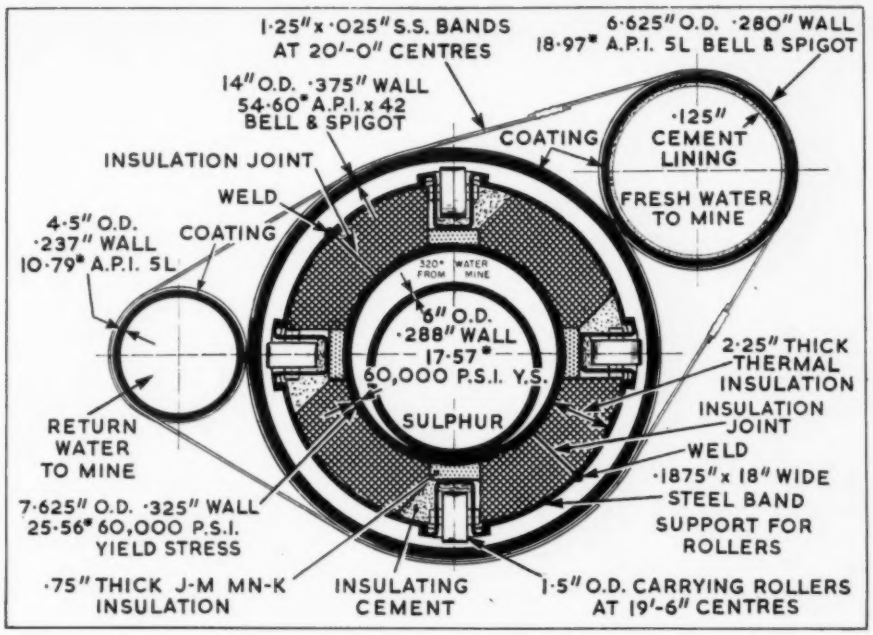

1·25" x ·025" S.S. BANDS AT 20'-0" CENTRES
6·625" O.D. ·280" WALL 18·97* A.P.I. 5L BELL & SPIGOT
14" O.D. ·375" WALL 54·60* A.P.I. x 42 BELL & SPIGOT
·125" CEMENT LINING
COATING
INSULATION JOINT
WELD
FRESH WATER TO MINE
4·5" O.D. ·237" WALL 10·79* A.P.I. 5L
COATING
320° FROM WATER MINE
6" O.D. ·288" WALL 17·57* 60,000 P.S.I. Y.S.
2·25" THICK THERMAL INSULATION
INSULATION JOINT
WELD
·1875" x 18" WIDE STEEL BAND SUPPORT FOR ROLLERS
SULPHUR
RETURN WATER TO MINE
7·625" O.D. ·325" WALL 25·56* 60,000 P.S.I. YIELD STRESS
·75" THICK J-M MN-K INSULATION
INSULATING CEMENT
1·5" O.D. CARRYING ROLLERS AT 19'-6" CENTRES

*Fig. 1 (right). Cross-section of off-shore sulphur pipeline at rollers.*

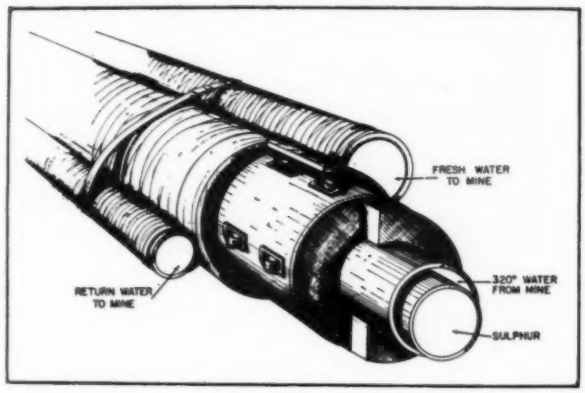



*Fig. 2 (below). Cut-away drawing of off-shore sulphur pipeline from the Grand Isle mine.*

supply of fresh water to the mine, but also provides additional supporting surface for the main pipeline in the soft clay at the Gulf bottom. The fresh water pumped through the second outside return line comes from the Mississippi River and is brought as ballast in the sulphur barges returning from Port Sulphur to the mainland base serving the off-shore Grand Isle mine. The quantity of fresh water brought to the mine in this way is sufficient only for domestic use and as general service water, the vast quantities of water necessary for the Frasch mining process being provided by heating sea water by a special process.

The fresh water delivered by the return pipe is not de-aerated and, because the dissolved oxygen makes it slightly corrosive, the line is protected inside by a coating of cement. The hot water used in the $7\frac{5}{8}$-in. pipeline for maintaining sulphur heat has to be of a very high quality to prevent corrosion and scale deposition.

Normally, pipelines exposed to varying temperatures, such as is the case of the Grand Isle mine pipe, are constructed with expansion joints or loops to overcome the thermal stresses. In the present case, the inner lines would normally expand about 55 ft when heated from 75°F to the 300°F operating temperature. In a buried line, expansion joints may not be effective and their cost was considered excessive, since by eliminating all expansion joints, construction costs could be greatly reduced.

To overcome the thermal stresses involved, a unique solution was adopted and this involved anchoring the line to the bed of the Gulf of Mexico so that the 55 ft of movement would not take place; but this meant that high tensile steel had to be used for the walls of the inner lines to give a yield strength of about 60,000 psi. Because the stress in the cooler 14-in. casing would be considerably less than in the hot inner lines, the use of Grade X-42 steel having a 42,000 psi yield was possible.

Before being buried, the outer casing was placed in 10,000 psi tension by exerting a pull on each end, and the ends were fixed to an anchor structure while the pipe was still in tension. This stress is relieved when the line reaches its operating temperature of about 110°F. Before the inner lines were placed in operation they were heated to about 225°F and allowed to expand their normal amount. While the pipes were in this expanded condition, each end of the inner lines was permanently fastened to the outer casing, which, in turn, had been anchored to the structures provided. Thus, when the inner lines were placed in operation and reached a temperature of 300°F, the thermal stress was compressive and the combined stresses were about 60% of the yield strength. Should the inner lines be cooled down to atmospheric temperature, the thermal stress would be replaced by tension and the combined stresses would amount to about 70% of the yield strength. In this way longitudinal movement of the pipeline has been practically eliminated and the use of expansion joints avoided.

When the pipeline has settled in its bed at the bottom of the sea and the soil has become remoulded around the pipeline, its resistance is expected to be sufficient then to prevent appreciable movement without the need for the anchoring structures which were regarded only as temporary facilities. At the shore end the line is buried for more than two miles in fine sand and no movement is expected there. At the mine end, however, it is buried in very soft clay which, it is anticipated, will yield somewhat to the pipeline stresses and allow a small amount of longitudinal movement.

ACKNOWLEDGMENTS

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# COPPER SULPHATE BY A CONTINUOUS PROCESS

Processes for the manufacture of copper sulphate pentahydrate crystals are reviewed. The older processes are essentially intermittent batch processes and their disadvantages are compared with the continuous process, some details of which are given for a comparable production

by F. MOLYNEUX

COPPER sulphate pentahydrate, or blue vitriol as it is known commercially, is the most important industrial chemical compound of copper and although it is employed in the preparation of a range of copper compounds, its main use is in agriculture for the control of plant diseases. It is an ingredient of Bordeaux mixture and it is used in small quantities by individual farmers, who make up a solution as required, plus the required quantity of lime.

By reason of the close connection with the agriculture of the large vine and potato growing countries of the world, copper sulphate has always been an important export chemical of both this country and the U.S.A., as the following figures show.

| | | 1954 | 1955 | 1956 |
|---|---|---|---|---|
| U.K. | Production Copper Sulphate Tons | 39,193 | 39,171 | 47,992 |
| U.S.A. | Production—1945. Production in short tons | | | |
| | copper refineries .. .. | 32,900 | | |
| | Other plants .. .. | 92,600 | | |
| | Total | 125,500 | | |
| | Total Exports | 34,967 | | |

The pentahydrate has been exported for agricultural use since 1885, and the usage and form has become therefore largely traditional. Thus the demand is mainly for firm, solid crystals, over $\frac{1}{2}$ in size. Crystals must be of at least 99.0% purity and possess a good colour; any trace of white crystal showing dehydration or greenish tints indicating high iron or nickel is highly undesirable.

The pentahydrate is generally sold in grades based on crystal size; these usually include large crystals (plus $\frac{1}{2}$ in. mesh), small crystals (minus $\frac{1}{2}$ in. mesh) and superfine or snow. There is in addition a small demand for pulverised crystal largely for manufacture into other copper compounds. Due to the traditional usage, the large crystals command a slightly higher price.

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## Economics of Copper Sulphate Manufacture

The prices of copper sulphate are generally based on the ruling price for copper metal at the time of sale, with an additional charge for acid and processing costs. This makes the production of copper sulphate alone uneconomical, and its main advantage lies in its being produced as a by-product, where the overheads and processing costs can be shared with other plants. Its production is useful because certain other valuable metals are released in the wastes from the process, such as tin, lead, nickel, and to a lesser extent, silver and gold.

Unfortunately, since the main usage is agricultural, the trade is largely seasonal, and to accumulate sufficient stocks for the demand requires that the processing be very flexible.

There is an economic hazard in carrying large stocks of the material. This is because the selling price of the sulphate is closely tied to that of copper which is itself a raw material with wide price fluctuations. To have a manufacturing plant that is flexible and capable of rapid increases in production, is therefore important. It is also necessary that in such a plant the amount of copper in process at any time should be small.

Since the price is based on the copper content at the ruling copper price, it is essential that service costs and handling charges, labour, etc., should be a very small fraction of the works cost.

## Manufacturing Methods

Originally, copper sulphate was produced in this country and in the U.S.A., as a by-product of the copper refineries. This formed the basis of the initial production of copper sulphate in this country[1] from nickel copper sulphide ore refining at Clydach in S. Wales.

In the Canadian process ore was first roasted in the open, then mixed with slag from the converter and smelted in a blast furnace. The product was then transferred to a converter mixed with flux, and oxidised with an air blast; the iron was removed in the slag leaving a mixture of nickel and copper sulphides to be sent to the refinery. Here the sulphides were roasted to oxides which were then heated with hot dilute sulphuric acid, to dissolve out almost the entire content of cupric oxide, the major part of the nickel

oxide being left behind. The nickel oxide was then converted to metal by the Mond Carbonyl process.

The resulting copper sulphate solution was heavily contaminated with iron and required careful air blowing under controlled pH conditions followed by filtration in order to obtain the requisite purity demanded by the trade; this filtration step was a troublesome one. Owing to the transfer of the electrolytic copper refineries to the immediate vicinity of the copper ore source (e.g. the copper belt of Rhodesia) this process is now no longer used for copper sulphate crystal, and a large proportion of U.S. copper sulphate production and the entire production in this country is by chemical companies who rely upon various scrap and waste copper residues. These copper residues are fire-refined, and the copper is preformed into shot at the refining stage by running the molten metal in a thin stream into water wells. The quality of the shot is largely dependent on that of the residue being refined but generally the two types of shot are produced: 1. Black shot containing appreciable quantities of impurities, mainly lead, tin and nickel; 2. Bright shot, comprising mainly copper with much smaller quantities of the above impurities.

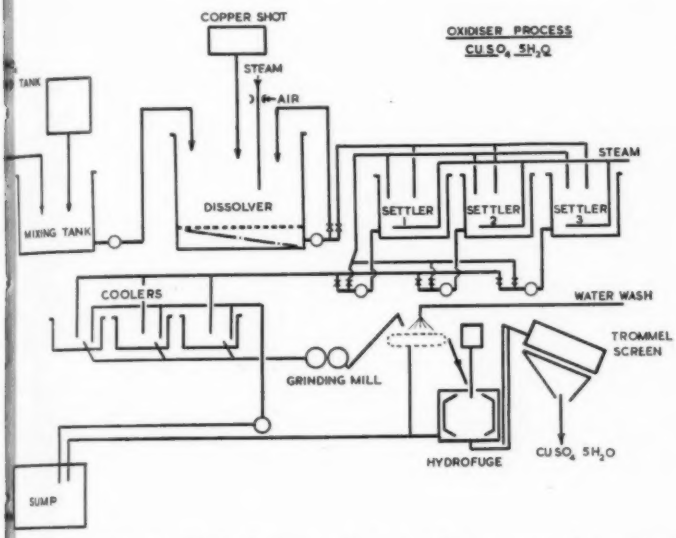


*Fig. 1. Flow diagram of the oxidiser process for copper sulphate manufacture.*

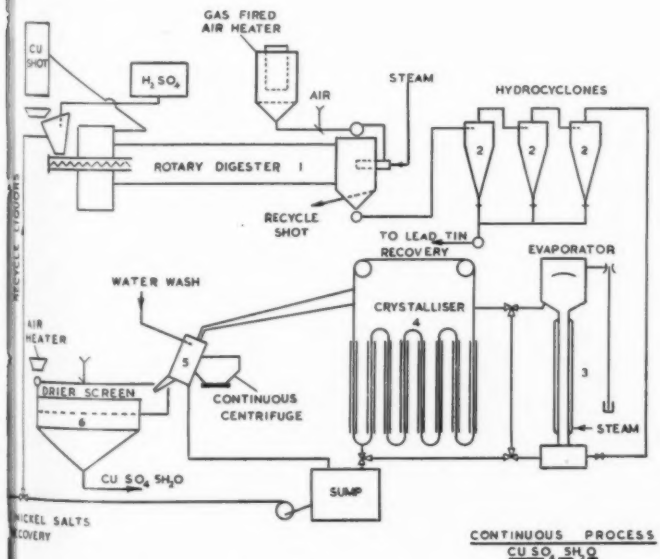


*Fig. 2. Process for the continuous production of copper sulphate pentahydrate.*

The fire-refining process is expensive and with the increasing supplies of good electrolytic grade copper scrap cheaper methods of preparation of copper for use in copper sulphate manufacture have been investigated. For example pulverising in attrition and ball mills is now being used.

This is important because the extraction of the impurities in the preformed scrap can take place chemically in the production of the sulphate and thereby help to reduce the cost of processing. In addition the finer state of subdivision greatly accelerates the rate of oxidation and solution of the copper in the sulphuric acid.

The principal process used both in this country and in the U.S.A. by chemical companies manufacturing copper sulphate crystal is the oxidiser process[2, 3, 4] and this process will be first outlined, together with its main difficulties.

The continuous process has been operated on a small scale occasionally to produce special products.

An outline of the items of plant which have been used is given in the flow sheet (Fig 1) and the features of such a plant to produce 500 tons per week of hydrated crystal are discussed below.

## The Oxidiser Process

The process is essentially a batch process and to ensure continuity of production it is essential to provide a multiplicity of units for each phase of the conversion, and to run these units on a carefully regulated cycle.

Thus the dissolver is filled manually with either bright or black copper shot which is subjected to oxidation by a series of blast pipes running vertically through the filled dissolver tank. These blast pipes are fed from steam ejectors with a mixture of live steam and air. The steam serves to entrain and convey ailr to the bottom of the shot for percolation through the bed; in addition condensation of a portion of the steam on the shot raises the temperature to 140-160°F and dilutes the acid used for conversion to sulphate with steam condensate. This latter must be allowed for in the acid strength fed to the dissolver, otherwise it might be difficult to attain, without evaporation, a solution sufficiently concentrated for crystallisation.

Dilute sulphuric acid mixed with weak mother liquor, is fed from the mixing tank to the dissolvers, and in percolating through the bed of oxidised shot dissolves off the oxide layer and takes this into solution as cupric sulphate.

Since the solution stage is much more rapid than the oxidation stage the acid solution is drained periodically from the tank in process, and this tank then reverts to the oxidisation cycle. It is therefore essential to have more than one dissolver in operation so that the cycle can proceed.

The dissolver is fitted with a false floor constructed in perforated acid-proof tiles and the acid mixture is circulated until the gravity tests show that the correct concentration has been reached (Fig. 1).

A considerable portion of the impurities, mainly lead and tin, remain in the dissolver tank and eventually so clog the shot that the tank must be dug out manually and the insoluble sulphates cleaned off the shot. The remainder of the insoluble impurities is in a finely divided suspension which can be washed through the floor with the liquor stream. These impurities are settled out as a slime in a series of heated settling tanks. In addition to raising the temperature to assist in rapid settling of the heavy tin lead slimes, these tanks give some evaporation and therefore concentrate the liquor prior to crystallisation.

Unfortunately, the convection currents caused by the evaporation, cause a portion of the finely divided slime to

be retained even after considerable periods, and it passes forward into the coolers, where it causes contamination of the bottom crystals. The heavy slimes accumulate in the settlers and are periodically dug out and passed to the recovery plant for lead tin recovery.

The concentrated liquors from the final settler are pumped forward to the coolers or crystallisers where the pentahydrate crystallises out over a period of some 10/14 days, solely under atmospheric cooling, evaporation being prevented by an occasional spray of water over the surface of the liquor in the cooler. Some crystallisation takes place on a series of lead straps suspended from timber spars above the cooler but a large proportion of the crystal is formed as a hard interlocking mass on the sides and the bottom of the vessel. The crystal must be broken up and removed manually from the cooler; the entire "pull" of crystal is then carted to the crusher to break up the mass for efficient extraction of entrained mother liquor and insoluble impurities. The washed crystal is then centrifuged, partially dried by a current of warm air, and finally screened through a trommel screen into the two main grades, plus oversize and undersize. It is essential to pack as soon as possible after screening, as otherwise the pentahydrate will effloresce and be converted to the trihydrate.

This process suffers from the following disadvantages.

1 Reaction rates are slow and therefore for moderate scale production a number of large reactors are required with large retention times.
2 Rate of production is not controllable so that in order to provide flexibility of production a multiplicity of units are required.
3 The process is intermittent and requires careful phasing to preserve continuity.
4 The process requires much labour and is difficult to mechanise. Barker[2] states that 7 process men are required for a production of 50 tons/week and due to the batchwise nature of the process requiring multiplicity of units for proportionate increases in production, the labour requirements are virtually proportionate to the production requirements. Thus for 500 tons/week a total of 50 men would be required.
5 Although the plant required is very simple due to the considerable amount of manual handling, maintenance of the lead lined equipment is high.
6 Floor space and process inventory are very high.
7 Crystal crop is aggregated and even with crushing and washing it is difficult to remove occluded impurities. A considerable amount of recycling is therefore necessary.
8 Service requirements, especially steam and pumping power are high, mainly through the extensive recycling and the inefficient utilisation of the steam inherent in the process.

These disadvantages render the process so inefficient that profitability margin is slight. Wide fluctuations in raw material costs can therefore render the production uneconomic.

*Solution Control*

| Solution to | Copper g/l | Iron g/l | $H_2SO_4$ g/l |
|---|---|---|---|
| Dissolving tank 27°Be at 52.5°F | 23 | 1.5 | 269 |
| Settling tank 34°Be at 72°F | 80 | 2.0 | 100 |
| Precipitating tank 39°Be at 80°F | 113 | 3.0 | 198 |
| Sump 21°Be at 33°F | 27 | 2.2 | 118 |

Gravity of mixture to dissolving tank—28°Be at 52°F.

A higher sp. gr. may be obtained by the use of more acid but an excessive quantity will not increase the rate of solution of the copper and will give a product of porous crystals filled with dilute acid. Oxidation and solution will not take place with sufficient rapidity until the concentration is 10 g/l Cu, 250 g $H_2SO_4$ per litre. (Shot heated to 180-200°F.)

## Continuous Process

The continuous process because of its greater flexibility, small process inventory, and reduced handling charges, is more efficient, particularly if the tonnage requirements are such that a moderately sized plant can operate continuously.

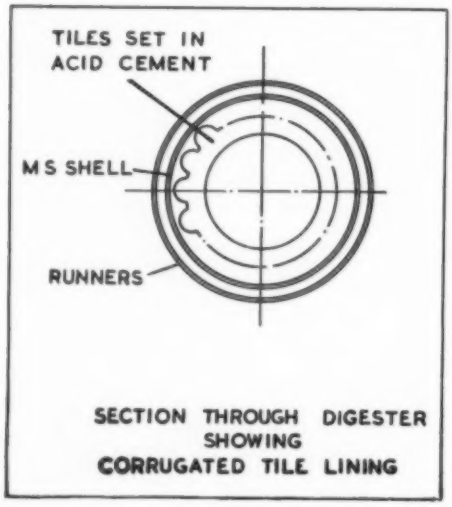


*Fig. 3. Cross-section through digester, showing corugated tile lining.*

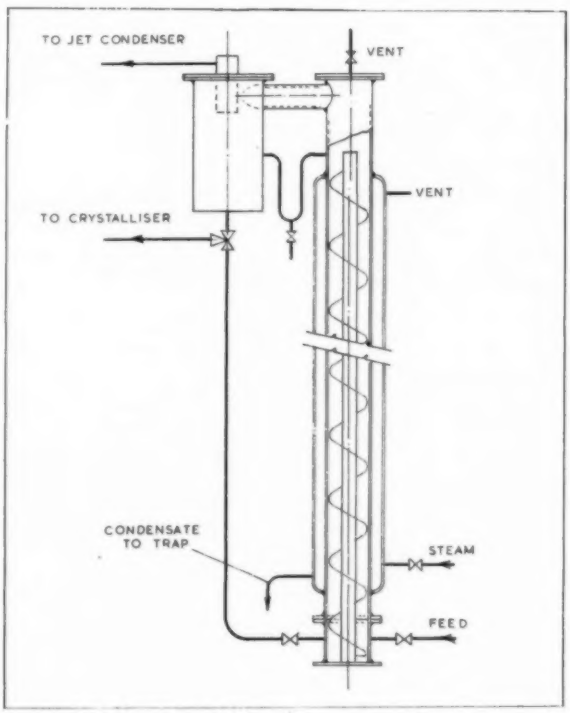


*Fig. 4. Climbing film type evaporator for copper sulphate solution.*

In order to operate with these objects in mind, it is essential to dissolve and crystallise continuously. Both these operations have been carried out on small scale plant for the production of 10/tons/week crystal. The data obtained could be used to scale up the plant for an estimated 500 tons/week production.

The plant items required are shown in Fig. 2; their design is now discussed.

*Dissolver or Digester*

The reactions occurring in this vessel are simultaneous oxidation of the preformed copper, either as shot or ground scrap or residues, together with solution of the oxide formed as sulphate in the weak sulphuric acid mixture. Preformed copper and sulphuric acid are fed continuously into a rotary tubular reactor. The reactor consists of a mild steel shell, lined with corrugated acid tiles set in acid cement (Fig. 3). The firing hood and the feed hood are similarly constructed in acid brick.

The feed of preformed copper is via a worm feeder constructed in Mo/Ti stainless steel and the feed pipe for the sulphuric acid mix is similarly constructed in either Mo/Ti or Monel.

The air for oxidation is preheated to a temperature of 800°F in an oil-fired preheater. Due to the possible explosion hazard which might result from hydrogen produced by the direct attack of sulphuric acid on the preformed copper, the air is preheated in an externally fired combustion chamber surrounded by the air heater space. The hot air is blown into the digester at a temperature of 800°F at a velocity and pressure sufficiently high to prevent blow-back to the combustion chamber. It is doubtful whether the saving of fuel by recirculating a portion of the exhaust air from the digester to the preheater is justified in view of the explosion hazards which might arise; the warm waste gas is therefore led to atmosphere via the preformed copper shot bunker. This forms a perfect entrainment separator for any acid mist which might possibly be entrained in the air stream meanwhile preheating and oxidising the copper.

Due to the lengthy retention period required in the dissolver (5/6 hr) the speed of rotation is very low—1 rpm—but this ensures that the minimum amount of copper is discharged with the sulphate solution from the rotary digester.

The discharge is passed through a stainless screen to the suction of a high head slurry pump constructed of either stainless steel or monel. The solution to be pumped is at 120°F and has the following concentration. 20 g/l Cu 250 gpl $H_2SO_4$.

From the digester the solution at the controlled concentration but containing in suspension small particles of undissolved copper and insoluble sulphates of tin and lead is pumped via the separators to the climbing film evaporator.

*Separators*

These are three 2 ft diameter hydrocyclones arranged in series.

They are fabricated from resin bonded fibreglass, a material used very satisfactorily at these temperatures and easy to maintain. The separated slurry is drawn from the base of the first separator, and is pumped to the lead and tin recovery plant for recovery of these metals, together with others such as silver which may be present.

In this process the copper sulphate is stripped from the waste and is returned for reprocessing. The clarified stream of copper sulphate solution from the last of the series of separators is then fed to the evaporator.

*Evaporator*

This is a single tube steam jacketed climbing film type evaporator (Fig. 4). Condensation of the evaporated vapours takes place in a jet condenser which also subjects the evaporator to a mild vacuum of 10-in Hg.

The concentrated solution at an outlet temperature of 180°F and 0.41 mass fraction $CuSO_4$ is fed to the crystalliser inlet.

*Continuous Crystalliser*

This provides a retention time of 5 hours. The concentrated copper sulphate solution is fed at a temperature of 180°F to the inlet of this water cooled crystalliser.

The cooling water and hot solution are fed in parallel flow in order to initiate nucleation from solution by shock cooling, thereafter the recirculated cooling water flow is carefully adjusted to give the maximum growth of the crystal on the nucleae formed and to minimise the formation of new crystals.

The growing crystals are conveyed through the cooling solution on perforated conveyor flights made of hard rubber with hard rubber flight links, the relative velocity between flight and solution being very low (1 ft/sec) but sufficient to provide agitation to eliminate big concentration gradients (Fig. 5).

The crystal is elevated from the mother liquor in the continuous crystalliser into a feed hopper feeding a continuous hydro centrifuge. The crystal is washed in the hydro centrifuge the liquors being returned with mother liquor from the crystalliser to the mixing sump where it is blended with fresh acid for feed to the rotary digester.

The washed and spun crystal is then dried with a current of warm air as it is being elevated to the trommel screen by a stainless steel wire mesh inclined elevator conveyor. The crystal is sized continuously in a rotating trommel screen into the three commercial grades, and is packed automatically into 1 cwt × 5 ply bitumen lined paper sacks.

Provision is made at the crystal silos for the drawing-off crystal required in special packs.

## Service and Labour Requirements

Good supplies of cooling water are required for process, preferably artesian well or borehole water. Low pressure steam at 25 psi should be used on the evaporators.

The only power usage is in drives to pumps, digester and crystalliser centrifuge and screens, and is very low.

Labour for the operation of the plant for a production of 500 tons/week is estimated at 5 men per shift including labour for packing but not for warehousing or dispatch. It is estimated that the continuous plant would be economical for production as low as 100 tons/week on a continuous 24-hour basis.

For feed liquor at 12°F the evaporator efficiency is 0.56, and steam requirements for evaporation are 25.500 lb/hr at 25 psig.

## Thermochemistry and Process Variables

In the Oxidiser process, the process in the dissolver is the oxidation of the static bed of copper shot followed by the solution of this oxide film in the spray of dilute sulphuric acid.

It has been suggested that this might be a three stage process with the intermediate formation of cuprous sulphate. The cupric sulphate in solution acts on the copper to form cuprous sulphate which is then oxidised by dissolved oxygen to the cupric state. This is seemingly the action by which dilute sulphuric acid attacks copper plant since the rate of attack is increased by a factor of 30 if the sulphuric acid solution be aerated at the same conditions of temperature and pressure.

In the oxidiser process itself however the formation of an oxide film is the first stage and is the controlling factor

in the rate of reaction. The mechanism of this heterogeneous reaction seemingly takes place in four stages.

(1) Diffusion of oxygen through the liquid film to the copper surface;
(2) Chemi-absorption of oxygen;
(3) Desorption as $Cu_2O$ or CuO or both;
(4) Solution of these products from the surface.

Since the reaction is heterogeneous, the rate will depend on the extent of the copper surface. The rate will therefore be greater for small particles than for large under otherwise equal conditions but it will increase logarithmically as the solution of cupric sulphate proceeds, due to the decrease in particle size and the corresponding increase in area.

The controlling stages in the reaction are 2 and 3, and as a consequence the rate of oxidation is enormously influenced by temperature and by surface reactivity. In the oxidiser process it is difficult to control either of these factors, since the reaction proceeds in a static bed at relatively low temperatures.

The reaction has, however, been carried out in a rotary kiln at much higher temperatures and the reaction time thereby reduced by a factor of $2 \times 10^3$.

These controlling reactions can be written:

1 $\begin{cases} 2\,Cu_2O + O_2 = 4\,CuO - 4300 \text{ cals/gr mol.} \\ 4\,Cu + O_2 = 2\,Cu_2O - 38{,}500 \text{ cals/gr mol.} \end{cases}$

The following reactions can be written for the further stages:

2 $\begin{cases} 2\,Cu_2O + H_2SO_4 = 2\,Cu_2SO_4 + H_2O \\ 2\,Cu_2SO_4 + O_2 = 2\,CuSO_4 + 2\,CuO \\ 2\,CuO + 2\,H_2SO_4 = 2\,CuSO_4 + 2\,H_2O + 20{,}000 \\ \text{cal/g mol.} \end{cases}$

Since reaction 1 is controlling, the design of digester should be based on the rate of this reaction.

Cupric sulphate pentahydrate crystallises from solution as large blue triclinic crystals with sp gr = 2.28. On heating this crystal it changes to the trihydrate with sp gr = 2.66 at 110°C changes to monohydrate sp gr = 3.20 and at 150°C changes to the anhydrous form as a greenish white crystalline powder. On further heating to 650°C this breaks down to cupric oxide with sp gr = 3.60.

The solubility curve of the pentahydrate is shown in the diagram (Fig. 6).

Since the solubility changes linearly with temperature it is usual to crystallise from a hot concentrated solution without evaporation by simple atmospheric cooling. This results in large crystals of non-uniform size, since the rate of crystallisation is completely uncontrolled.

Uniform crystals can be obtained by a controlled crystallisation but the rate of growth from a hot concentrated solution is somewhat slow and as a result considerable liquor retention time must be allowed to grow the plus ½ in demanded.

Nuclei are easily formed *in situ* but careful control is necessary to grow on these and prevent "false grain".

An improved method for the production of copper sulphate has been investigated in Russia.[5] These investigations have been so successful that this "continuous" process is rapidly replacing the old batch process referred to in this article.

Fundamentally this process is the old tower process which the author had experience of operating on a small plant producing 10 tons/week of copper sulphate in the period 1953/54 and amply verified the Russian statement that throughput can be doubled using this type of plant. This tower process has found considerable favour in Sweden using large reinforced concrete towers and feeding copper shot to the tower top by means of skip hoist.

The reaction gas used is hot moist $SO_2$ gas with an $SO_2$ content of 10/12%. The process does not however give the improvement in reaction rate of the magnitude claimed for in the continuous process outlined above, mainly because the process is not strictly speaking a continuous "flow" process: the bed of copper shot is static giving loss of flexibility and accompanying hold-up of material inventory, both of which are serious disadvantages with a product of this type.

Investigations of the mechanism of reaction made in this country have clearly established that the formation of copper sulphate is via an electro-chemical action as distinct from the older chemical diffusional process proposed by Stepin.

These newer theories replace those based on diffusion of oxygen to the metallic surface by conduction of cations and electrons to the oxygen surface.

Thus when copper is oxidised $Cu^{++}$ ions move to the copper surface where they cause copper to dissolve as $Cu^+$ ions according to the following

$$Cu^{++} + Cu \rightarrow 2Cu^+$$

Then $Cu^+$ ions move to the oxygen surface where they give up electrons to form $Cu^{++}$ and $Cu_2O$

$$4Cu^+ + O \rightarrow 2Cu^+ + O^{--} + 2Cu^{++} = Cu_2O + 2Cu^{++}$$

Because of the electro-chemical nature of this rate controlling process it is possible by the use of the methods outlined to obtain the greatly increased reaction rates so necessary in a continuous flow process.

## Materials of Construction

In the intermittent process for the manufacture of copper sulphate crystal the materials of construction comprise timber, lead, copper and acid-proof concrete.

The dissolvers, settlers and mixing tanks consist of timber framed vessels, panelled in precast acid-resisting concrete and lined with 12-lb lead sheet.

Pumps used are of the open impeller centrifugal pattern with impeller and shaft in Mo/Ti stainless steel and the case in chemical lead. The chemical lead casing is not very resistant to the abrasion from undissolved shot, insoluble sulphates and deposited crystals. At the points where these conditions are likely to be met as well as higher temperatures, it has been found advantageous to employ a stainless steel liner.

Copper can be used with advantage where no possibility of accelerated attack due to aeration can take place and where some possible advantage due to its ductility and high conductivity can arise; thus it has been used in crystallisation coolers. It has, however, a slight disadvantage in that it is not as easily maintained in plant and equipment by the usual methods though bronze welding has proved satisfactory for most repair work with some evidence of a decrease in corrosion resistance probably due to galvanic action.

In a continuous plant with the more severe conditions of aeration, temperature and abrasion the best construction materials are acid-proof ceramic tile and Mo/Ti stainless steel. For lower temperatures the newer plastics such as plastic-bonded glass-fibre and even polythene are used successfully as they have excellent abrasion and corrosion resistance. Stainless steel, unless of the Mo/Ti variety, will not resist abrasion and corrosion of weak sulphuric acid solutions at moderate temperatures under aerated conditions. Wrought iron and silicon iron have been proposed but their use introduces a serious risk of iron contamination.

### *Process Control*

The main points of control are on the gravity and composition of the liquor from the digester. Initially until the reaction has got under way the rate of production of liquor is much less than under steady conditions. The maximum amount of hot air is required and the liquor is recirculated around the digester until balanced conditions have been achieved. Similarly tin, lead slurries are not drawn from the base of the hydrocyclones initially, tests being made to find

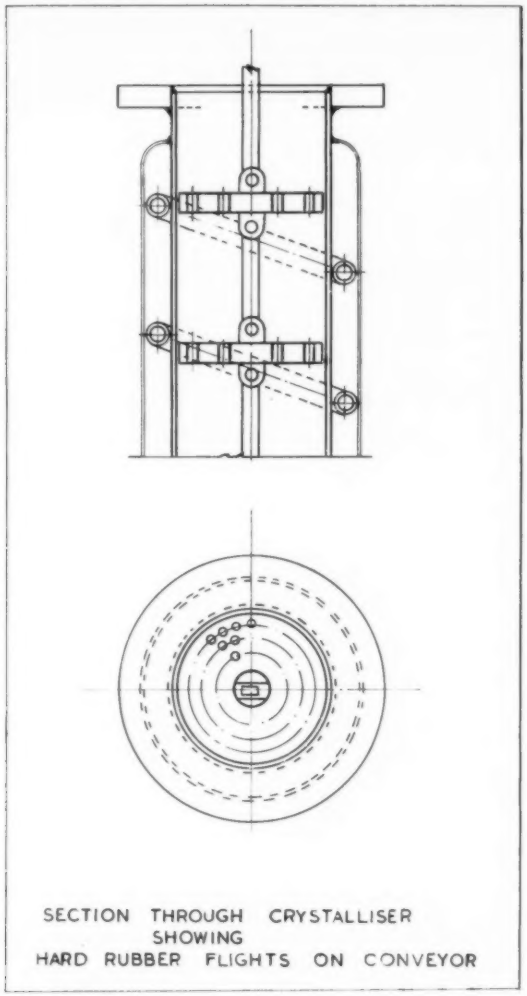


*Fig. 5. Plan and cross-section of crystalliser, showing perforated hard rubber flights on conveyor.*

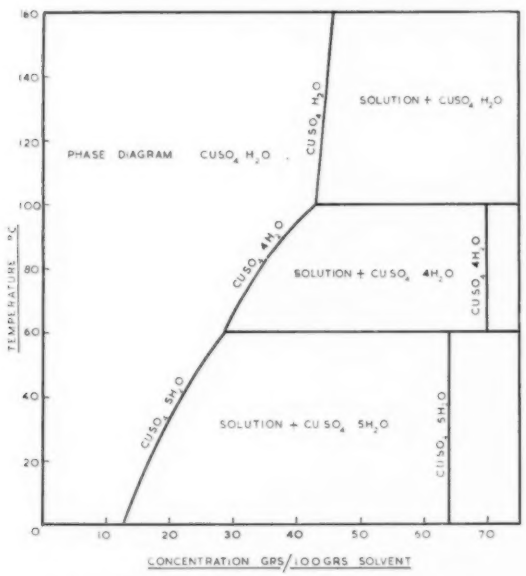


*Fig. 6. Copper sulphate water phase diagram.*

the extent of the deposition prior to pumping over to recovery. Once balanced conditions have been achieved then slurries can be pumped over continuously. The recirculated mother liquors from the crystallisers are analysed for the concentration of soluble sulphates of nickel, zinc, lead and tin and once these have attained proportions approaching solubility limits the liquors are withdrawn and evaporated for further sulphate (mainly nickel) recovery.

Crystal purity is tested regularly for arsenic, tin, lead, nickel, zinc, manganese and iron, these being the main impurities carried forward from the raw material.

*By-product recovery*

Tin lead slurries are evaporated down and calcined in a rotary furnace to complete the oxidation of any remaining copper. The copper content is then extracted with sulphuric acid and the sulphate liquors returned to process. The tin lead sulphates are then converted to carbonates. Tin-lead is extracted from these carbonates by reduction. Mixed concentrated sulphates by-passed from the re-circulated mother liquor are treated for recovery of nickel sulphate.

The process generally consists of salting out the nickel sulphate as nickel ammonium sulphate $NiSO_4 (NH_4)_2 SO_4 6H_2O$ from the acid solution of mixed sulphates. The neutral mother liquors are then passed through a sump filled with scrap iron to remove copper residue; the iron replacing copper in solution as iron sulphate; the liquor contains sulphates of ammonia, zinc, tin, lead and iron.

*Future Prospects*

Copper sulphate shares with soda ash and sulphuric acid the distinction of being one of the oldest inorganic chemicals in industrial production. Although the demand fluctuates according to the price of copper, in bulk, copper sulphate is consistently third in the list of exports following soda ash and caustic soda.

Whilst copper sulphate has an important use as the starting point for the production of a whole range of copper compounds, including combinations of copper sulphate with organic acids for emulsifiers, oil dopes, impregnating compounds, the main future outlet for copper sulphate is largely in the field of agricultural chemicals.

Most of the copper sulphate produced in this country is exported for use in the traditional form as Bordeaux mixtures, but expanded application can be foreseen in the dusting of agricultural crops. Its future use may be either in combination as solid with commercial fertilisers or as a solution in methanol in conjunction with the newer ranges of pesticides.

The recognition of copper as a trace element in the nutrition of plants has established copper sulphate as an essential part of the formulation of any fertiliser for preventing or alleviating deficiency symptoms.

This is particularly important where large tracks of virgin country are being opened up for purely arable cultivation as in Russian Siberia and even where the soil is not lacking in copper applications of copper sulphate have been found to increase yields or improve the quality of crops.

It is clear, however, that if copper sulphate is to maintain and improve its position as a leading bulk chemical it can only be done on a basis of increased efficiency in manufacture. This requires a concentration of production in continuous units.

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6.063.8

# THE SCALE-UP OF AGITATED LIQUID-LIQUID SYSTEMS IN DISSIMILAR VESSELS

The author derives an equation which relates mean droplet diameter to the other variables relevant to the scale-up of agitated vessels for liquid-liquid dispersions such as are encountered in emulsion and suspension polymerisations. The expression derived circumvents the strict adherence to the conditions of geometric and dynamic similarity required by the conventional theory of mixing.

by B. A. HILLS

## Introduction

THERE is comparatively little information in the literature upon the agitation of two immiscible liquids. In this field only a few quantitative attempts have been made to correlate mean drop diameter with the parameters of state relevant to such systems. Even these are restricted to the conditions of geometric and dynamic similarity consistent with the general theory of mixing.

They contain two principal approaches:

(a) The older, as expounded by MILLER and MANN,[1] in which mean drop diameter is associated with the power input per unit volume.

(b) The more recent, as expounded by VERMEULEN, WILLIAMS and LANGLOIS[2] and concisely summarised by JOHNSTONE and THRING,[3] in which mean drop diameter is related to the Weber number.

However, for the small company scaling-up an emulsion or suspension polymerisation, it is seldom possible to comply with the similarity conditions essential to the above approaches. Nevertheless, for reasons of final mean particle size and of inter-phase rates of transfer of monomer or reaction initiator, it is usually necessary to adhere closely to the required initial drop-size distribution.

This paper is an attempt to derive a mathematical expression relating mean drop diameter to the other variables encountered in the scale-up of agitated systems for emulsion and suspension polymerisations in dissimilar reaction vessels.

## Method

Approaching the subject from first principles, the following procedure will be adopted:

(i) Listing all variables likely to influence drop-size in a stirred vessel.

(ii) Complete dimensional analysis to determine the variables upon which drop-size is dependent, subsequently expressing their functionality in terms of dimensionless groups.

(iii) Obtaining the required comprehensive relationship by integration of the general equation incorporating the partial differentials supplied by the single-variable expressions quoted in the literature.

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**Symbols Used**

$d$ = mean particle diameter;
$D$ = impeller diameter;
$N$ = impeller speed;
$\mu$ = bulk liquid viscosity;
$\rho$ = bulk liquid density;
$\sigma$ = interfacial energy per unit area;
$P$ = power input;
$\Pi$ = power input per unit volume;
$\theta$ = the diameter ratio;
$L$ = dimension of length;
$M$ = dimension of mass;
$T$ = dimension of time;
Re = Reynolds number;
Fr = Froude number;
$N_p$ = Power number.

$K$, $K'$, $K''$ and $K'''$ are dimensionless constants.
$k$ and $k'$ are constants.

| *Variable* | *Symbol* | *Dimensions* |
|---|---|---|
| Mean particle diameter | $d$ | $L$ |
| Impeller diameter | $D$ | $L$ |
| Impeller speed | $N$ | $T^{-1}$ |
| Bulk liquid viscosity | $\mu$ | $ML^{-1}T^{-1}$ |
| Bulk density | $\rho$ | $ML^{-3}$ |
| Gravitational density difference of phases | $\Delta\rho.g$ | $ML^{-2}T^{-2}$ |
| Surface energy per unit area | $\sigma$ | $MT^{-2}$ |
| Power input per unit volume | $\Pi$ | $ML^{-1}T^{-2}$ |

Thus $d = f[D, N, \mu, \rho, (\Delta\rho \,.\, g), \sigma, \Pi]$

or $d = \varphi\,[D^{\alpha}, N^{\beta}, \mu^{\gamma}, \rho^{\delta}, (\Delta\rho \,.\, g)^{\epsilon}, \sigma^{\lambda}, \Pi^{\omega}]$

where $\varphi$ now represents linear functionality.

Completing the dimensional analysis in the normal manner:

$d = \varphi[D^{(1-2\gamma-\epsilon-3\lambda-\omega)}, N^{(-\gamma-2\epsilon-2\lambda-3\omega)}, \mu^{\gamma}, \Pi^{\omega}, \rho^{(-\gamma-\epsilon-\lambda-\omega)}, (\Delta\rho \,.\, g)_{\epsilon}, \sigma^{\lambda}]$.

Hence
$$\frac{d}{D} = \varphi\left[\left(\frac{D^2 N\rho}{\mu}\right)^{-\gamma}, \left(\frac{D\rho N^2}{\Delta\rho \,.\, g}\right)^{-\epsilon}, \left(\frac{N^2 D^3 \rho}{\sigma}\right)^{-\lambda}, \left(\frac{\Pi}{D^2 N^3 \rho}\right)^{\omega}\right] \quad \ldots . (1)$$

Thus the expression can be reduced to one in dimensionless terms, these being:

$\left(\frac{d}{D}\right) = \theta_d$ — the diameter ratio.

$\left(\frac{D^2 N \rho}{\mu}\right)$ = Re — the Reynolds Number.

$\left(\frac{D N^2 \rho}{\Delta \rho . g}\right)$ = Fr′ — analogous to the Froude number (Fr) = $\frac{D N^2}{g}$

$\left(\frac{N^2 D^3 \rho}{\sigma}\right)$ = We — the Weber number.

$\left(\frac{\Pi}{D^2 N^3 \rho}\right) = N_\pi$ — analogous to the Power number $(N_p) = \frac{P}{D^5 N^3 \rho}$

$$\text{Thus } (\theta_d) = \varphi'[(\text{Re}), (\text{Fr}), (\text{We}), (N_\pi)] \quad \ldots . (2)$$

where $(N_\pi)$ accounts for power input;
(We) accounts for interfacial disturbance;
(Re) accounts for local turbulence;
(Fr′) accounts for vertical mixing throughout the bulk of material.

Expressing Equation (2) in terms of partial differentials:

$$d(\theta_d) = \frac{\partial(\theta_d)}{\partial(\text{Re})} . d(\text{Re}) + \frac{\partial(\theta_d)}{\partial(\text{Fr})} d(\text{Fr}) + \frac{\partial(\theta_d)}{\partial(\text{We})} . d(\text{We}) + \frac{(\theta_d)}{(N_\pi)} . d(N_\pi).$$

$$\text{i.e. } d(\theta_d) = \frac{\partial(\theta_d)}{\partial(N_\pi)} \cdot \frac{\partial(N_\pi)}{\partial(\text{Re})} . d(\text{Re}) + \frac{\partial(\theta_d)}{\partial(N_\pi)} \cdot \frac{\partial(N_\pi)}{\partial(\text{Fr})} . d(\text{Fr}) + \frac{\partial(\theta_d)}{\partial(\text{We})} . d(\text{We}) + \frac{\partial(\theta_d)}{\partial(N_\pi)} . d(N_\pi)\ d(\theta_d) = \frac{\partial(\theta_d)}{\partial(\text{We})} . d(\text{We}) + \frac{\partial(\theta_d)}{\partial(N_\pi)} \left\{ d(N_\pi) + \frac{\partial(N_\pi)}{\partial(\text{Re})} . d(\text{Re}) + \frac{\partial(N_\pi)}{\partial(\text{Fr})} . d(\text{Fr}) \right\} \quad \ldots . (3)$$

Now for fully baffled, or correctly off-centred mixers, the power factor $(N_\pi)$ is independent of the Froude number $(\text{Fr})^3$ where kinematic, geometric and dynamic similarities are preserved.

$$\text{Thus } \frac{\partial(N_\pi)}{\partial(\text{Fr})} = 0 \quad \ldots . (4)$$

According to the work of RUSHTON, COSTICH and EVERETT,[4] for values of (Re) above 300:

$$(N_\pi) = K(\text{Re})^m,$$

$m$ being given in this reference for various ranges of (Re) for dynamically similar systems.

$$\text{Thus } \frac{(N_\pi)}{(\text{Re})} = Km(\text{Re})^{m-1} \quad \ldots . (5)$$

Combining Equations (3), (4) and (5), we have:

$$d(\theta_d) = \frac{\partial(\theta_d)}{\partial(\text{We})} . d(\text{We}) + \frac{\partial(\theta_d)}{\partial(N_\pi)} [K(\text{Re})^{m-1} d(\text{Re}) + d(N_\pi)] \quad \ldots . (6)$$

According to VERMEULEN *et al.*[2]
$(\theta_d) = K''(\text{We})^{-0.6}$ for dynamically similar systems.

$$\text{Thus } \frac{\partial(\theta_d)}{\partial(\text{We})} = -0.6K''(\text{We})^{-1.6} \quad \ldots . (7)$$

No quantitative data could be found for the variation of $(\theta_d)$ with $(N_\pi)$, so an approach will be taken analogous to that of RITTINGER in his calculation of the power to disintegrate solids.

[Power proportional to energy of new surface formed.]

The many anomalies found in Rittinger's Law should not deter from the use of the suggested analogy to liquids, since their prime cause (crystal lattice imperfections rendering it difficult to estimate effective surface area) is no longer operative.

Thus power $(P) \propto \Sigma a$ [$a$ = drop surface area].
i.e. $P = n\bar{a}$ [total of $n$ drops].
Now $\bar{a} \propto d^2$ [$V$ is total volume].
and $n \propto \frac{qV}{d^3}$ [$q$ is volume fraction of disperse phase].

Thus power per unit volume $(\Pi) = \frac{P}{V} \propto \frac{q}{d}$

Since $(N_\pi) = \frac{\Pi}{D^2 N^3 \rho}$,

we have for a two-phase system of fixed proportions where other factors are kept constant:

$$(\theta_d) = \frac{d}{D} = \frac{K' D^6 N^3 \rho}{N_\pi} = \frac{K' \mu^3}{\rho^2} \frac{(\text{Re})^3}{(N_\pi)}$$

Since density ($\rho$) and viscosity ($\mu$) are constant for perfect scale-up:

$$\theta_d = \frac{K'''(\text{Re})^3}{(N_\pi)}$$

$$\text{and } \frac{\partial(\theta_d)}{\partial(N_\pi)} = -K'''(\text{Re})^3 . (N_\pi)^{-2} \quad \ldots . (8)$$

Combining Equations (6), (7) and (8) we have:

$$d(\theta_d) = -0.6K''(\text{We})^{-1.6} d(\text{We}) - K'''(\text{Re})^3 (N_\pi)^{-2} mK(\text{Re})^{m-1} d(\text{Re}) + d(N_\pi) \quad \ldots . (9)$$

But $K'' = (\theta_d)(\text{We})^{-0.6}$,
$K = (N_\pi)(\text{Re})^{-m}$,
and $K''' = (\theta_d)(\text{Re})^{-3}$,

Thus Equation (9) thus gives the required equation for general integration:

$$\int \frac{d(\theta_d)}{(\theta_d)} = -0.6 \int \frac{d(\text{We})}{(\text{We})} - \int \frac{d(N_\pi)}{(N_\pi)} - m \int \frac{d(\text{Re})}{(\text{Re})}$$

giving $\log(\theta_d) = -0.6 \log(\text{We}) - \log(N_\pi) - m \log(\text{Re}) +$ constant.

$$\text{Thus } (\theta_d) = k(\text{We})^{-0.6} . (N_\pi)^{-1} . (\text{Re})^{-m} \quad \ldots . (10)$$

However, in most mixers, $\text{Re} \simeq 10^5$
But for $\text{Re} > 10^4$, $m = 0$[2]
Hence Equation (10) reduces to:

$$(\theta_d) = \frac{k(\text{We})^{-0.6}}{(N_\pi)} \quad \ldots . (11)$$

where $K$ is a dimensionless constant characteristic of the reaction mixture.

## Discussion

In accordance with general principles of scale-up, it should be emphasised that the whole of this work refers to initial liquid mixtures of identical composition. Thus, in the primary choice of variables, the selection of impeller diameter ($D$) and speed of rotation ($N$) is sufficient to define completely the equilibrium state of any such system conforming to conditions of geometric and dynamic similarity.

For example, for the same mixture composition in a vessel of fixed dimensions, two immiscible liquids will always display identical drop-size distributions for the same impeller speed and the corresponding power consumption. Now, if only the vessel geometry is changed, but not stirring rate, then both size distribution and energy consumption will alter—illustrating that power must be used as a parameter, no longer synonymous with impeller revs., in defining the system.

Thus, by introducing the further variable ($\Pi$) to account for power consumption ($P$) and total liquid volume ($V$), and specifying fully turbulent flow conditions, we reduce the number of degrees of freedom by one, or perhaps two. In defining these further parameters describing the mode of creating turbulence, and its lower limit, we are thus obviating the need for strict adherence to the similarity conditions im-

posed by conventional theory.

Hence Equation (11) should apply to the scale-up of agitation in any vessel where submerged baffles completely suppress vortex formation. Correct off-centring can have the same effect of reducing bulk toroidal movement, and such systems may be regarded as "fully baffled".[6]

To put Equation (11) in perspective by substituting primary variables:

$$d = \frac{k \,.\, D^{1\cdot2} \,.\, N^{1\cdot8} \cdot \rho^{0\cdot4} \,.\, V}{P} \qquad \left[\Pi = \frac{P}{V}\right]$$

[$k$ is dimensionless constant]

Hence for a system of given liquid composition:

$$d = \frac{k' \,.\, D^{1\cdot2} \,.\, N^{1\cdot8}\, V}{P} \qquad \ldots.(12)$$

[$k'$ is a constant for a given liquid-liquid system and has dimensions $M^{0.2} \,.\, L^{1.2} \,.\, T^{-3}$.]

Since Equation (12) should hold for all vessels, it should at least be satisfied by the results quoted for the particular case where similarity conditions are complied with.

Thus, for this case, we lose the advantage of our two extra degrees of freedom gained by the comprehensive nature of Equation (12), since we can take:

(i) $V \propto D^3$

(ii) $P \propto D^5N^3\rho$ (the simple single-phase power equation) and Equation (12) gives the approximate expression

$$d \propto D^{-0.8} \,.\, N^{-1.2}$$

which agrees with the suggested conditions of scale-up quoted by JOHNSTONE and THRING[3] of keeping $D^2N^3$ constant.

Thus Equation (12) is more comprehensive, yet in reasonable agreement with the results of cited literature.


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[3] Johnstone and Thring, "Pilot Plants, Models and Scale-up Methods", McGraw Hill, 178.
[4] Rushton, Costich and Everett, *Chem. Eng. Prog.*, **46**, 395.
[5] Rushton, *Chem. Eng. Prog.*, **47**, 485.


# Producing Pure Hydrogen from Dissociated Ammonia

THE new Norelco process produces pure hydrogen from dissociated ammonia gas by low-temperature condensation of nitrogen in the mixture followed by low-temperature adsorption of the residual nitrogen; it gives a product containing less than 100 ppm nitrogen impurity. The low-temperature refrigeration is supplied by a Norelco Cryogenerator. The unit was built by Cryogenerators Inc., a subsidiary of North American Phillips Co.

Dissociated ammonia feed gas, consisting of one-quarter nitrogen and three-quarters hydrogen by volume, is compressed in a non-lubricated compressor. The compressed gas mixture is cooled in a water-cooled heat exchanger and processed to remove residual traces of undissociated ammonia. The cleaned gas is filtered of adsorbent particles, and then passed into a feed heat exchanger to be cooled by the cold product streams.

The cold feed gas is then cooled in heat exchanger coils in order to condense out the maximum amount of nitrogen from the feed. The hydrogen gas-nitrogen liquid mixture is then passed into a separator to disentrain the liquid nitrogen.

Hydrogen gas is then passed through an adsorber to remove the residual nitrogen. The resultant pure product hydrogen gas is heated to ambient temperature in the feed heat exchanger. The purity of the product hydrogen is monitored by a hydrogen analyser with provisions for re-cycling the gas through the process if it does not meet the product specifications. The product hydrogen gas is made available directly for process use or for storage.

## Refrigeration Cycle

The recondenser comprises two pistons working in a single cylinder. Around the cylinder in annular rings are a heat exchanger and a regenerator in which heat is stored. The top of the cylinder is also surrounded by heat-exchange fins.

In operation, the pistons move together in the lower part of the cylinder and compress the working medium. Compressed gas flows out of the cylinder into the heat exchanger, where cooling water removes the heat of compression.

The compressed gas then flows to the regenerator and gives up heat to the cold porous cells. From here, the chilled medium passes into the dome above the top piston. When this piston is drawn downward, the gas expands rapidly and its temperature plunges.

At this point, nitrogen gas, passing over the fins outside the dome, is quickly condensed by the extremely cold medium. The latter is then pushed back into the regenerator by the upward movement of the piston. Here it absorbs the heat left earlier in the cycle, leaving the regenerator once more frigid. The medium then passes into the lower part of the cylinder, between the two pistons, and is ready for another cycle.

## Hydrogen Production

Dissociated ammonia, after compression to 600 psi, passes through an adsorber which removes any undissociated product. It then flows to the unit which contains all the low-temperature equipment where it is cooled to −275°F by the cold product streams.

Intense cold comes in the next section, where a series of three heat exchangers chills the feed with liquid nitrogen boiling at −310°F (atmospheric pressure), −330°F (250 mm Hg abs.) and −345°F (50 mm Hg abs.). The stream, now mostly liquid nitrogen and gaseous hydrogen, passes through an entrainment separator and an adsorber which remove the nitrogen.

The cold, purified hydrogen (final product) then absorbs heat from the incoming feed stream until it is within 5 deg. of ambient temperature.

Liquid nitrogen that drains from the hydrogen-nitrogen separator is expanded to lower pressure in another separator. Dissolved hydrogen flashes off during the expansion, to be combined with another small hydrogen stream described below.

The adsorbers, which strip the final nitrogen from the pure hydrogen, work in tandem—when one is on stream, the other is regenerated by a stream of pure hydrogen.

Low-pressure nitrogen vapours from vacuum exchangers are exhausted through the feed heat exchanger, pressurised to 5 psi, and split into two streams. One part serves as a purge on the cold box to keep out air and moisture. The other goes back into the system through the recondenser which converts it back to liquid.

The system includes all necessary pressure-relief valves, gauges and thermocouples, and liquid-level controls needed to control the process.

Liquid nitrogen is used throughout the cold box to immerse the separators, adsorbers and heat exchangers.

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# ADSORPTION IN INDUSTRY

A report of a recent symposium.* The papers presented deal with the following topics: the use of activated charcoal for treating industrial effluents; a process control chromatograph of special design for refinery analyses; the principles of adsorption; and the use of molecular sieves

## Introduction to Adsorption *by J. W. ARMOND*†

THIS paper dealt with the basic principles of adsorption. At the outset the author explained the term "sorption", which is used where the nature of the phenomenon is uncertain or where a general term is required. Then followed an account of effect of pressure and temperature on adsorption and the methods of representing adsorption equilibria, the isotherm, isobar and isostere were shown. In discussing the isotherm more fully, the "classical" or "Freundlich" adsorption isotherm was introduced followed by LANGMUIR's treatment for monolayer coverage which was shown to reduce to the classical isotherm. The principal characteristics of the four types, Van de Walls or physical adsorption, chemisorption, activated adsorption and persorption, were described.

The BRUNAUER, EMMETT and TELLER (B.E.T.) treatment of multilayer adsorption was outlined and the relationship of their equation to the five known types of adsorption isotherm was discussed. The use of the B.E.T. treatment in determining the surface area and average pore size of an adsorbent from a measured adsorption isotherm was explained.

Adsorption was discussed from the point of view of the physical properties of the adsorbate, and evidence for the general rule that the most easily condensed and most soluble gases are the most readily adsorbed was given. It was shown that deviations from this rule may be expected where chemisorption or capillary condensation occur, also where the adsorbent is ionic in nature and where the adsorbate possesses a dipole moment, a quadrupole moment or is unsaturated. The preferential adsorption by molecular sieve adsorbents of molecules small enough to penetrate to the active centres of the adsorbent may also lead to departures from the rule.

Next followed brief accounts of the role of chemisorption in heterogeneous catalysis and adsorption from liquid solution. The principle of chromatography which was first recognised and applied in liquid solutions was then introduced.

The second part of the paper discussed dynamic adsorption processes from the point of view of chromatography. Chromotography can occur with a gas or liquid moving phase and a liquid or solid stationary phase. It was pointed out, however, that it is dependent on adsorption phenomena only where the stationary phase is solid and the moving phase gas or liquid. The three types of chromatography—elution development, displacement development and frontal analysis—were defined, and it was then shown qualitatitively how these three types of chromatography may operate in fixed-bed adsorption, moving-bed chromatography and the hypersorption process.

## The Use of Activated Charcoal in the Treatment of Trade Wastes

by D. H. SHARP‡

PARTICULARLY in view of recent legislation—the Rivers (Prevention of Pollution) Act of 1951 and the Estuaries and Tidal Waters Act of 1960, and the report of the Armer Committee[1]—effluent disposal is a very topical subject. There is a growing realisation that the effluent from a process is as important as the end-product.

Normally speaking, there are only two possible ways of disposing of a trade waste—either into a local sewer or into a water-course. In the one case, the Sewage Board has to be satisfied that it can receive and treat the effluent, or in the other case, the River Board will lay down conditions with which the effluent must comply. A sewage board or river board will normally lay down standards for:

(a) heavy metals and other inorganic constituents;
(b) dissolved solids;
(c) suspended solids;
(d) oxygen demand.

Two tests are commonly employed to estimate oxygen demand: oxygen absorbed from permanganate, usually called the O.A. or P.V., which is a measure of the inorganic or chemical reducing power, and the Biochemical Oxygen Demand or B.O.D. This is a measure of the dissolved oxygen removed from natural water after a standard period of time (usually five days) at a standard temperature (usually 68 or 70°F) under other standard conditions. Thus an effluent of high B.O.D. may rapidly remove the dissolved oxygen from a water-course so that the fish cannot breathe.

Methods available for the treatment of trade wastes include, chemical treatment (e.g., neutralisation; precipitation of heavy metals); physical processes (sedimentation, microstrain-

* Graduates' and Students' Section, Institution of Chemical Engineers, Symposium on Adsorption in Industry, London, October 20 and 21, 1960.

The accounts of MR. RUHEMANN'S paper on gas drying and MR. HAGGER'S on carbon bisulphide recovery have been held over for publication in a later issue.

† Gas Separation Department, British Oxygen Research Development Ltd.
‡ Fisons Ltd., Harvest House, Felixstowe, Suffolk.

TABLE I—Harston Factory Products

| Common Name | Chemical Composition |
|---|---|
| *Weedkillers* | |
| DNC | 3:5 dinitro-o-cresol |
| Dinoseb | 2:4 dinitro-6-sec-butylphenol |
| MCPA | 2-methyl-4-chlorophenoxyacetic acid |
| 2:4.D | 2-4 dichlorophenoxyacetic acid |
| MCPB | 2-methyl-4-chlorophenoxybutyric acid |
| TCA | trichloracetic acid |
| CMU | 3-p-chlorophenyl-1:1 dimethyl urea |
| *Insecticides* | |
| DDT | 1:1:1 trichloro-2:2 di-(p-chlorophenyl) ethane |
| Parathion | 00-diethyl 0-p-nitrophenyl thiophosphate |
| Schradan | octamethyl pyrophosphoramide |
| dimefox | bisdimethylamino-fluorophosphine oxide |
| *Fungicides* | |
| COC | copper oxychloride |
| — | phenyl-mercury chloride |

TABLE II—Effluent Constituents

| Product Manufactured | Compounds Present in Effluent |
|---|---|
| Weedkillers | DNC,* dinoseb,* MCPA, 2:4.D, OSBP†, MCPB, TCA, CMU, cresol,‡ phenol,‡ chlorinated cresols‡ and phenols,‡ nitro and nitroso compounds,* glycollic acid,‡ sodium chloride,‡ various solvents and formulants. |
| Insecticides | DDT, parathion, schradan, dimefox, substituted amine phosphates,‡ solvents and formulants, sodium chloride,‡ sodium sulphate.‡ |
| Fungicides | Copper salts, phenyl-mercury salts. |

* Although present in relatively low concentration, not affected by biological treatment.
† Intermediate compound o-sec-butylphenol, present in small quantities. The remainder of the substances normally present in very small concentrations.
‡ Present in fairly high concentration.

TABLE III—Effect of pH on Adsorption

| Analytical Results (ppm) | | Untreated Effluent | pH of Influent to Charcoal | | |
|---|---|---|---|---|---|
| | | | > 12 | 7.2 | 3.0 |
| Phenols | a | 79 | < 0.4 | < 0.4 | < 0.4 |
| | b | 14.4 | < 1.0 | < 1.0 | < 1.0 |
| Dinitro cresol (DNC) | a | 180 | < 1.0 | < 1.0 | < 1.0 |
| | b | 30.0 | < 1.0 | < 1.0 | < 1.0 |
| Total colour (as DNC) | a | 280 | 133 | 70.4 | 14.7 |
| | b | 134 | 105 | 2.7 | 1.5 |
| Glycollic acid | a | 985 | 947 | 380 | 756 |
| | b | 218 | 210 | 129 | 155 |
| Secondary amines as dimethylamine | a | 20.0 | 2.4 | 18.8 | 19.0 |
| | b | 392 | 40 | 168 | 315 |
| Permanganate value (4 h) | a | 1096 | 517 | 141 | 94 |
| | b | 1472 | 1036 | 453 | 143 |

a Results from percolate of 7 litres.
b Results from percolate of 9 litres.

TABLE IV—Typical Analytical Results

| All analyses in ppm | Crude Effluent | After Charcoal Treatment | As Discharged to River |
|---|---|---|---|
| Phenols | 800 | 8 | 0.8 |
| Dinitro-ortho-cresol | 200 | 1 | 0.5 |
| Colour (as DNC) | 500 | 7 | 3 |
| Glycollic acid | 1000 | 800 | 25 |
| Permanganate value (4 h) | 1500–2000 | 300 | 30 |
| 5-day BOD | 2500 | 400 | 20 |

TABLE V—Charcoal Consumption

| | |
|---|---|
| Granular material (30 B.S.S. mesh) | |
| Charcoal turn-round (over 3 years) .. .. .. .. | 1472 tons |
| Charcoal purchased to maintain stocks .. .. .. | 159 tons |
| "Fines" (30 B.S.S. mesh) .. .. | |
| Total accumulated .. .. .. .. .. .. | 92.5 tons |
| Unusable (passing 100 B.S.S. mesh) .. .. .. .. | 7.5 tons |
| Usable material (30–100 B.S.S. mesh) .. .. .. | 85.0 tons |
| Net usage = 159 − 85 .. .. .. .. .. .. | 74 tons or 5.0% of turn-round |

ing); and either aerobic or anaerobic biological treatment. Biological methods are cheapest in operation if not in first cost, and form the principal treatment at sewage works. The strength of effluents is normally related to crude domestic sewage and treatment costs are assessed accordingly.

Adsorption forms a useful adjunct to these methods of treatment. Adsorption treatment is usually more expensive than the methods commonly employed; applied to effluent treatment, adsorption is essentially a complementary treatment, and, if activated charcoal is used as adsorbent, a very versatile treatment method is available. The use of adsorption is particularly valuable in cases in which the effluent contains substances which are bactericidal, i.e., substances which would preclude normal biological oxidation treatment, substances which can be biologically oxidised only with difficulty, or when it is necessary to reduce the polluting load given to a biological filter system in order to meet a stringent condition for treated effluent.

## Development of Activated Charcoal

Two forms of activated charcoal have been developed—steam-activated and chemically-activated charcoal. A good outline of steam-activated charcoal has been given by FIELDNER, HALL and GALLOWAY[2] and of chemically activated charcoal by TROMP and MATTHEE.[3] Chemically-activated charcoal is usually in powder form and employed for liquid-phase applications. It is usually discarded after use. Steam-activated charcoal is usually in granular form and is employed for vapour phase applications. Such charcoal is usually recovered and reactivated when "spent".

## Characteristics of Liquid Phase Adsorption

As compared with adsorption in the vapour phase, liquid phase adsorption is characterised by:

(i) Low sorptive power of the adsorbent—only about a quarter of vapour phase adsorptive capacity is normally exhibited by a given adsorbent when employed in the liquid phase.

(ii) Slow adsorption rate. In the liquid phase, diffusion is the rate determining factor. The present author[4] has shown from the Van't Hoff isochore $d/dT \log K = \triangle H/RT^2$, where $K$ is the adsorption velocity constant and $T$ the absolute temperature, that $\triangle H$ is the heat of diffusion of water in water.

From simple diffusion theory it follows that adsorption rate varies as the square of geometric surface of the adsorbent.

(iii) Character of adsorbate. Electrolytes are feebly adsorbed; undissociated molecules are adsorbed more readily than ions. Hydrophilic molecules are less readily adsorbed than hydrophobic molecules.

(iv) pH of solution. Since free acids are adsorbed more strongly than salts, acidic compounds are adsorbed more effectively in acid solution and basic compounds more readily in alkaline solution.

(v) Concentration of solution. From the Freundlich isotherm $x/m = ac^{1/n}$, it follows that $m_1/m_2 = (V_2/V_1)^{1/n}$, where $m_1$ and $m_2$ are weights of adsorbent required to adsorb the same quantity of adsorbate from volumes $V_1$ and $V_2$ of solution. In practice, this implies that adsorption treatment should be introduced when solutions are as strong as possible. The use of adsorption as a final mopping-up operation, as sometimes proposed in effluent treatment, should be avoided.

(vi) Character of adsorbent. The adsorbent should be chosen to provide the optimum relating to granule size, adsorptive capacity, hardness, and price.

In sugar refining is found an old-established industry employing liquid phase adsorption on a substantial scale. The adsorbent commonly employed in the U.K. is bone-char. Two cisterns of bone-char are employed in tandem; the char is reactivated on site and is reused. The substances adsorbed are of large molecular weight and are hydrophobic: since the sugar is hydrophylic, the impurities are selectively adsorbed.

Applied to effluent treatment the principles are much the same. That is to say, essentially vapour phase adsorption techniques are employed in the liquid phase. In practice it may not be economic to reactivate charcoal on site, but to return it to the manufacturers for this service. Transport is, of course, an important cost if this is done.

## Fisons Pest Control Effluent Treatment Plant

The factory is at Harston, almost on the banks of the River Cam, five miles upstream from Cambridge. Very stringent conditions are required for the discharged effluent and only limited dilution water is available.

Typical products of the Harston factory are given in Table I. Many of the products are bactericidal or bacteriostatic in character. The effluent constituents are shown in Table II. The substituted phenols cannot be biologically treated in strong solution. The dinitro compounds pass through a biological filter system unchanged.

Activated charcoal treatment is therefore employed for three reasons:

(i) to adsorb bactericidal compounds;
(ii) to adsorb dinitro compounds;
(iii) to reduce concentration of substituted phenols to a level at which they are susceptible to biological oxidation.

## Adsorption System

The method adopted is similar to that of the sugar-refining industry—adsorption in cisterns or towers, rather than admixture and filtration. A hard, granular steam-activated charcoal is employed of mesh size 10-22 B.S.S. mesh.

It was found to be much easier to handle the charcoal in the form of a slurry, to fill and empty the towers, than to handle dry granular material. A downwards flow through the bed was also adopted. It was found that channelling was avoided by this means; if channelling should occur, the fault is remedied by "back-washing", i.e., reversing the flow so as to lift the bed and allow it to resettle.

Originally it was intended to carry out one stage of the adsorption under acid conditions, so as to improve the adsorption of the acidic compounds present. Table III shows the result of a laboratory experiment demonstrating the effect of pH on adsorption. Thus the second bank of three towers was installed with rubber lining. In the event, the policy of acidic adsorption was abandoned, mainly due to the difficulty of reactivating charcoal saturated with acid, and adsorption is carried out under neutral conditions. However, the rubber lining to the towers has proved to be beneficial, as it has improved the life of the structure against abrasive attack.

The two rows of three adsorption towers employed at Harston are shown in Fig. 1. Each tower is 7 ft 6 in. in diameter and 25 ft high overall and capable of holding 7-10 tons of activated charcoal. The adsorbent is supported normally on 30 mesh gauze between perforated mild steel plates; the liquid flow is normally 3000 gal/hr, being controlled by a float switch in the top of the tower. A system of lines, pumps and valves is provided, enabling any required combination of towers to be employed.

When a tower has become spent it is emptied, in slurry form, into the deslurrying bunkers alongside. These are constructed of acid-resisting brickwork; the ends are closed by removable gauze doors and the contents of the tower are allowed to deslurry by drainage, the supernatant liquor passing back to crude effluent storage.

## Charcoal Reactivation

It was established in the laboratory that a temperature of 600-700°C in a current of steam was necessary for effective treatment. Fig. 2 shows the charging end of the standard rotary oil-fired furnace employed. It will be seen that the feed is co-current with the flame, so that the gases desorbed from the charcoal pass over a hot bed of material before reaching the stack. By this means the stink nuisance, otherwise a very real one, is avoided. Feed to the furnace is through two scoops affixed to the barrel, as shown. Control of atmosphere is essential and a good precombustion chamber materially assists in attaining good atmosphere control. The discharge end of the rotary furnace is shown in Fig. 3. The curved discharge tube serves as an air seal and heat exchanger so that the charge is cooled somewhat before leaving the furnace. The charge is further cooled, passed over normally a 30-mesh vibrating screen to remove fines, and is then elevated into storage hoppers, from where the charge is reslurried and fed back into the towers. A certain amount of buffer storage is provided, but most of the charcoal storage is done in the actual towers.

The factory effluent to be treated is made alkaline at collection

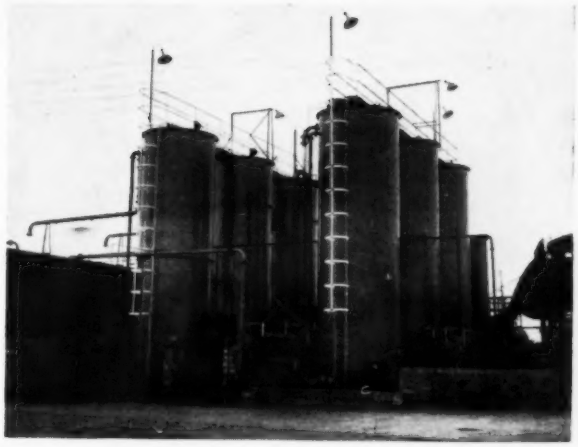

*Fig. 1. Adsorption towers for the treatment of liquid effluent at Harston; each tower holds 7-10 tons of activated charcoal. Liquid flow is normally 3000 gph.*

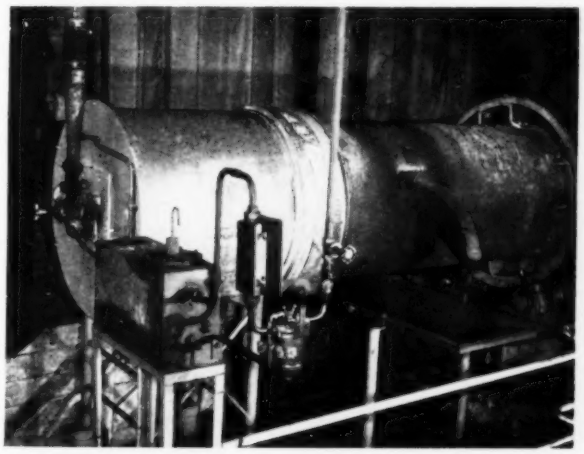

*Fig. 2. Rotary reactivation furnace—feed end. This provides a temperature of 6-700°C in a current of steam.*

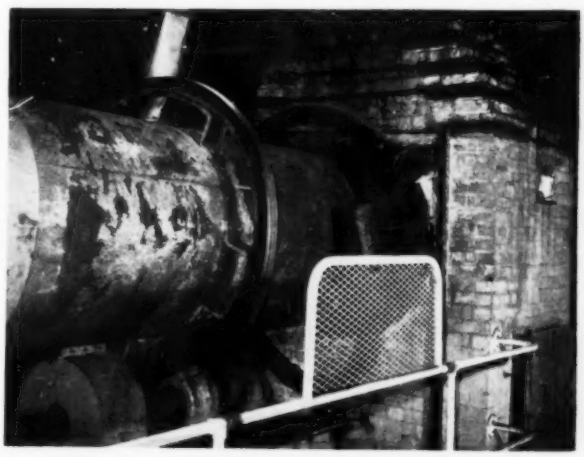

*Fig. 3 Rotary reactivation furnace—discharge end. The curved discharge tube serves as an air seal and cooler.*

and is pumped through cast-iron pipes to the effluent treatment site. Here it is bulk stored in sectional steel tanks. The pH is automatically adjusted to neutrality by sulphuric acid addition and adsorption carried out at pH 7. After charcoal treatment the effluent is stored in more storage balancing tanks. This intermediate storage is necessary in order to operate the adsorption at maximum efficiency, the system being operated until the liquor in these tanks is such that the biological treatment system that follows will just produce a discharge of adequate quality.

The complete effluent treatment plant has been previously described.[5] Plant efficiency achieved is given in Table IV. The substantial contribution made by adsorption treatment will be noted. The process has been operated for a sufficient period for reasonably reliable figures to be available on charcoal make-up; over the years a substantial stock of fines (through 30 B.S.S. mesh material) was accumulated. It was found that on rescreening on the 100 B.S.S. mesh the 30-100-mesh material could be used quite satisfactorily at a reduced flow rate. Table V shows the amount of charcoal make-up necessary after a substantial period of normal plant operation. A charcoal loss of 5.0% after three years' operation has been recorded.

ACKNOWLEDGMENTS

The author acknowledges indebtedness to several former colleagues at Fisons Pest Control Ltd., in particular to MR. A. E. LAMBDEN, with whom he has collaborated over a number of years. The paper is published by permission of the Board of Directors of Fisons Pest Control Ltd.

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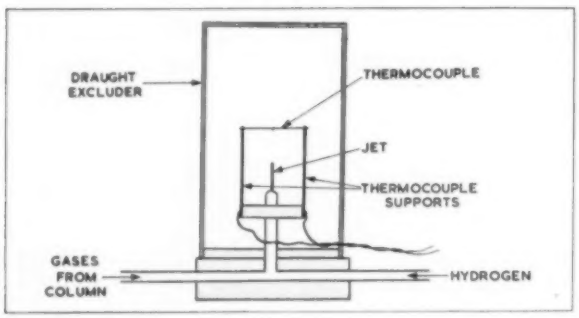



*Fig. 4. Hydrogen microflame detector which employs nitrogen as a carrier gas and in which the effluent from the column is fed.*

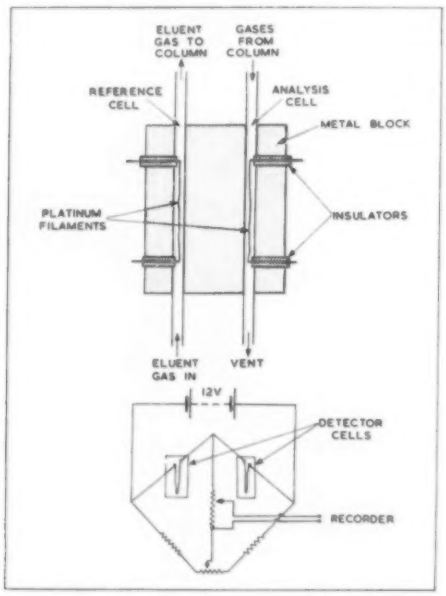



*Fig. 5. Katharometer detector. The instrument depends upon variations of carrier gas thermal conductivity as each peak is eluted.*

# Chromatography for Process Control

by C. P. WILLIAMSON*

MR WILLIAMSON stated that when the planning of the recent expansion of British Petroleum's Kent Refinery commenced in 1956, it was evident that the emphasis upon recovery of butanes and butenes from several processes for feeding to the alkylation unit, upon the purity of isobutane-rich streams within that unit and upon the purity of isopentane required as a blending component for aviation gasolines would require close attention to the analysis of various light hydrocarbon streams. Previously the necessary gas analysis had been carried out by the use of conventional low-temperature distillation equipment with an infra-red absorption spectograph for the determination of individual hydrocarbons in close cuts. This method was time-consuming, requiring up to 8 hr for complex mixtures, and a more rapid method was obviously desirable.

In chromatography was seen a technique eminently suitable for the analysis of refinery light hydrocarbon streams, which, although complex, were at least composed of known hydrocarbons. In addition, it offered a simpler approach than the mass spectrometer.

The available commercial instruments were more versatile than necessary for refinery use, and it was concluded that instruments designed for the special requirements of the refinery would be cheaper and more efficient. Accordingly, in collaboration with the B.P. Research Centre at Sunbury, the refinery staff developed and constructed several instruments, some for general application and others for specific analyses. Since 1957, all gas analysis of hydrocarbon streams has been carried out with these locally-made instruments.

## Basic Principles

The basic principle of the chromatographic separation of hydrocarbons is that if a mixture is first adsorbed on to a suitable *stationary phase* in a column and then a stream of eluting *carrier gas* is passed through the column, the individual constituents of the mixture will be separated and will appear in a definite order in the exit gas from the column. The intervals which elapse between successive *peaks* representing the components being eluted from the column are standard for a given column operated under given conditions of temperature and carrier gas type and flow, and are characteristic of the individual components.

In order to apply this principle, the sample must be introduced quantitatively, and it must be possible to detect and measure the peaks of individual components at the exit from the column.

* B.P. Refinery (Kent) Ltd.

*Fig. 6. Refinery standard apparatus. Left: Front view. Above, right: Analyser chassis top view showing two of the four detectors with draught shields removed. Below, right: Analyser chassis underside showing four columns.*

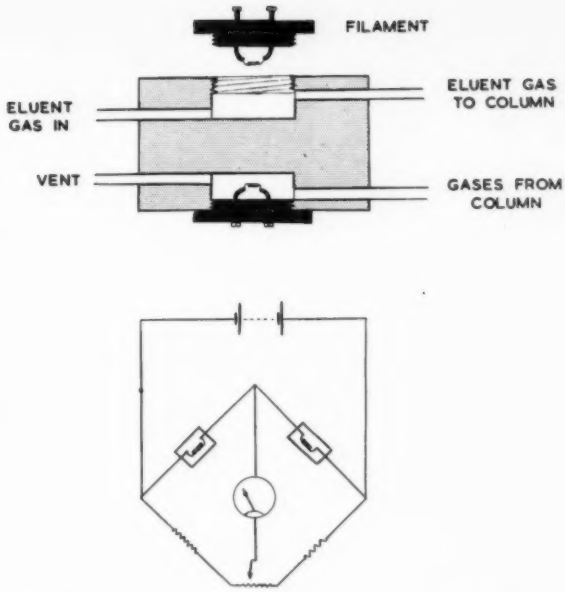



*Fig. 7. Platinum filament resistance bridge detector. The detector uses the well-known principle that hydrocarbons burning on a platinum filament raise its temperature and resistance.*

These are by no means the simplest parts of the problem of application, and on the resolution of difficulties in these sections depends the success of the method in any given application. There are, therefore, four principal factors to be considered.

A. Sample introduction.
B. Choice of column, stationary phase and carrier gas.
C. Detector system for peaks.
D. Presentation of results.

## A. Sample Introduction

Sample handing, involving the use of only microlitres of liquid and frequently less than 1 ml of gas, is a delicate matter. Liquid samples are measured and injected through serum caps by micrometer hypodermic syringes, although when small, repeatable volumes are required a very neat method is to use a short length of capillary tubing, preferably metal, as a constant-volume pipette. Gas samples are measured roughly by a constant-volume pipette built into a by-pass system at the column inlet. This system is very dependent upon system pressure measurement for its accuracy. The use of hypodermic syringes for the injection of gas samples has now been adopted as a standard. This technique is simple and particularly elegant when gas samples are handled in football bladders.

## B. Choice of Column, Stationary Phase and Carrier Gas

The choice of adsorbent to be used as the stationary phase is governed by the required separation. With the exception of silica gel, which is used for some specific analyses, most of the stationary phases consist of selected organic compounds adsorbed on "Celite' 'or powdered firebrick. Liquids used in the preparation of stationary phases include: dimethyl sulpholane; cetanes (standard diesel engine reference fuel); gas oil; silicone oil; squalane (hydrogenated shark-oil); dinonyl phthalate.

Each of these materials has particular characteristics and, as will be seen later, several different columns have been incorporated in the standard apparatus. By choice of column and carrier gas conditions, the separations can be made extremely sharply, and very sharp peaks obtained. For precise work involving higher boiling liquids, thermostatic control is achieved quite simply by an electrically heated liquid jacket round the coiled column. However, for most gas analysis work normal changes of ambient temperature in a large laboratory reasonably free from draughts have little or no effect.

These columns have been used with either air or nitrogen as carrier gas.

## C. Detector Systems for Peaks

The hydrogen micro-flame detector has been used most extensively in refinery apparatus, with nitrogen as a carrier gas. It is an extremely simple and sensitive device consisting (Fig. 4) of a small hydrogen flame, into which the effluent from the column is fed, burning under a precious metal thermocouple. To ensure steadiness the whole is surrounded by a shield and combustion air is supplied under slight but controlled forced draught. As each peak of separated hydrocarbon component is eluted into the flame, there is an increase of heat output and a response from the thermocouple. The normal output of the thermocouple is "backed off" electrically so that each peak is recorded above a steady baseline representing the normal condition of hydrogen and nitrogen only being fed into the flame. Another neat method of "backing off" is to arrange a differential flame detector in which the effluent from the colum is fed into one detector and the carrier gas only into another. The two detectors are carefully balanced so that any difference in E.M.F. is due solely to eluted components.

In addition to the hydrogen microflame, the katharometer (Fig. 5) and the platinum filament resistance bridge (Fig. 7) have also been used. It is a very sensitive detector and is used with inert gases such as nitrogen, helium and argon. For the immediate needs of the refinery, it offered little advantage over the proven work of the hydrogen flame detector and its use has not been extended beyond experimental instruments. On the other hand, the platinum filament resistance bridge was incorporated in a very simple apparatus for specific analyses, details of which are given below. This detector uses the well-known principle that hydrocarbons burning on the surface of a heated platinum filament will raise the temperature and the resistance of that filament. This is the basis of the commercial inflammable-gas-detecting instruments.

## D. Presentation of Results

The chromatogram which results from the use of a strip chart in conjunction with the hydrogen flame detector is, in essence, a series of areas each proportional to the heat liberated

by the combustion of a quantity of hydrocarbon. If the areas are computed, either by geometric means or weighing the paper area, the areas can be reduced to a volume of gas by the application of a factor: specific volume/heat of combustion. This gives the volume of gas represented by the peak in an arbitrary unit, and the sum of all the derived volumes is taken as the total sample volume for the calculation of individual mol per cent of components.

If the volume and conditions are carefully chosen, the peaks can be made very sharp and the relationship between area and peak height constant and reliably steady over long periods. In such cases the tedium of cutting and weighing peaks can be replaced by simple linear measurement and the use of a peak height factor in the calculation.

### Refinery Standard Apparatus

The standard apparatus constructed for refinery use consists of four units with four different columns provided (Fig. 6):

alumina for separation of hydrogen and the lower hydrocarbons;
cetane on firebrick for separation of saturated hydrocarbons;
dinonyl phthalate on firebrick for separation of normal butane, iso- and normal pentanes;
dimethyl sulphonane on firebrick for separation of saturated and unsaturated hydrocarbons.

### Butane-in-gasolene Analysis

For the control of a debutanising operation in the refinery, where a butane content of less than 0.2 per cent is specified, a very simple apparatus was devised. Air is used as a carrier gas and detection is by platinum filament resistance in a Wheatstone bridge circuit (Fig. 7). The display is on a milliammeter.

A measured volume of gasoline is injected by micrometer syringe and the height of the butane peak recorded. The instrument is calibrated with samples of known butane content, so that the milliammeter reading can be converted directly to an absolute amount of butane contained in the known sample volume. Using this technique, it is possible to adjust sample size to obtain a repeatable deflection on the milliammeter, and since the sample is measured accurately the whole chromatogram does not have to be obtained. Indeed, by reversing the air flow through the column after the peak has been measured, the rest of the components can be purged rapidly off the column in the reverse direction.

### Stream Monitors

Experience in the B.P. Kent refinery has been limited to a commercial instrument monitoring a normal butane stream for isobutane and isopentane. The recorded chromatograms, repeated at frequent and regular intervals, have enabled plant operators to make adjustments to the distillation train from which the stream is produced, in order to minimise loss of either isobutane or isopentane in that stream. The possibility of using the monitor in an automatic control system must be examined carefully before it is assumed to be practicable.

Even in the simple case quoted, a finite time must elapse between samples, and, although the introduction of improved detector systems and the great advances in the use of capillary columns with very high resolving powers have reduced the time elapsed for analysis, the method is still an intermittent one, requiring a "memory" and comparative function on the part of the analysing instrument, and an extrapolating function based upon experience. In a more complex system involving the analysis of a multi-component mixture, the "terms of reference" for a control instrument can be stated as follows: (a) Inject $x$ microlitres of sample accurately to a capillary column (where $x$ might be as little as 0.001). (b) Measure the peak heights of, say, 10 peaks. (c) Select the peak corresponding to the nominated hydrocarbon and "remember" its height in particular. (d) Automatically convert the 10 peak heights to quantities by use of 10 individual factors, summate and divide the quantity corresponding to the nominated peak by the sum. (e) Present this answer to an assessing circuit which will take action should the value differ from a preselected value. (f) Repeat as frequently as possible.

Such a controller will require a combination of extremely fine engineering for the sample introduction system, complicated electronics for the detection system, and computer techniques for the controlling system. These things are possible, but it will be necessary to ensure that the end result will result in greater economy or efficiency commensurate with the expense involved, especially when compared with the results which would be obtained from the intelligent use of a simpler monitor by the plant operator who would scarcely be superseded by the controller.

MR. WILLIAMSON concluded his paper by expressing his thanks to the chairman and directors of The British Petroleum Co. Ltd. for permission to publish it.

# The Industrial Use of Molecular Sieves

by J. F. EDWARDS*

### Introduction

MOLECULAR sieves are synthetic zeolite adsorbents. They differ from other adsorbents in that they are crystalline rather than amorphous and have pore openings of uniform dimensions. Adsorption takes place inside cavities to which these pores lead. Discussion demonstrated that molecules may be separated on the basis of molecular size degree of polarity and carbon bond saturation. Table I illustrates the adsorption characteristics of molecular sieves.

### Process Cycles

The process cycles listed below were described. In an actual process a combination may be utilised.

(1) *Thermal Swing Cycle.* This is defined as a cycle which employs different temperature levels for adsorption and desorption. An advantage is that high adsorbent loadings can normally be obtained, but the cooling step is an additional item of expense.

(2) *Pressure Swing Cycle.* Adsorption is carried out under pressure and desorption under reduced pressure. Tem-

TABLE I

| Adsorbed on 4A and 5A | Adsorbed on 5A but not 4A | Not Adsorbed on either 4A or 5A |
|---|---|---|
| Water | Propane and higher n-paraffins up to at least $C_{14}$ | Iso-butane and all iso-paraffins |
| Carbon dioxide | Butene and higher n-olefins | Iso-propanol and all iso-secondary, and tertiary alcohols |
| Carbon monoxide | n-butanol and higher n-alcohols | Benzene and all aromatics |
| Hydrogen sulphide | Cyclopropane | Cyclohexane and all cyclics with at least four-membered rings |
| Ammonia | Freon 12 | carbon tetrachloride |
| Nitrogen | | Hexachlorobutadiene |
| Oxygen | | Freon 114 |
| Methane | | Freon 11 |
| Methanol | | Sulphur hexafluoride |
| Ethane | | Boron trifluoride |
| Ethanol | | |
| Ethylene | | Molecules larger than 5.0 Angstroms |
| Acetylene | | |
| Propylene | | |
| n-propanol | | |
| Ethylene oxide | | |

* Chemicals Division, Union Carbide Ltd., 8 Grafton Street, London, W.1.
The term "Union Carbide" is a trade-mark of Union Carbide Corporation.

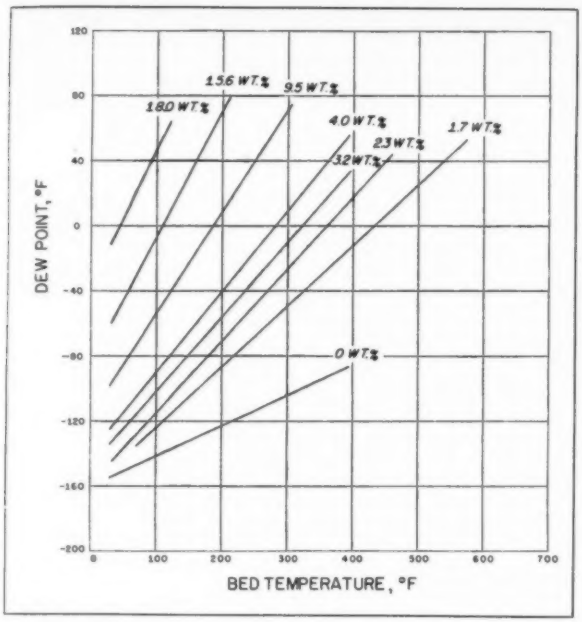



*Fig. 8. Minimum attainable dew point as a function of water loading and bed temperature (molecular sieve Type 5A).*

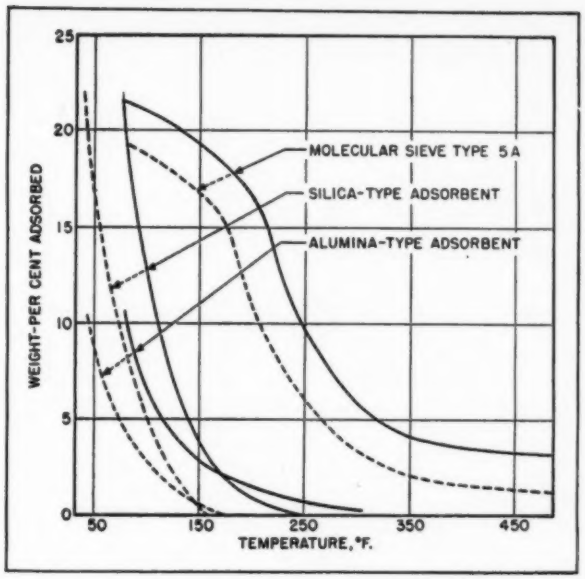



*Fig. 10. Water vapour adsorption isobars. Dotted lines show effect of 2 per cent residual water at the start of adsorption.*

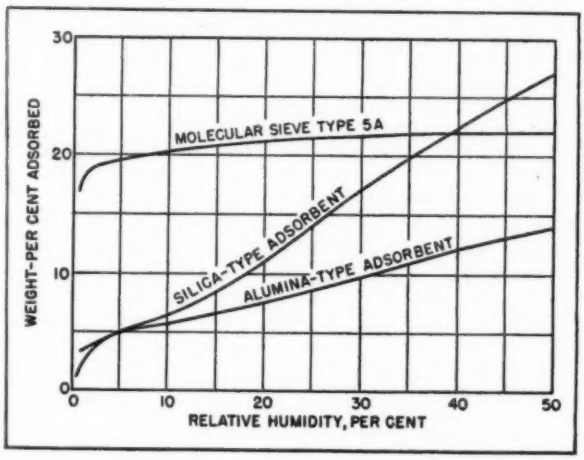



*Fig. 9. Water vapour adsorption at 25°C (equilibrium data). The graph indicates that molecular sieves have more than four times the drying capacity of other adsorbents at very low humidities.*

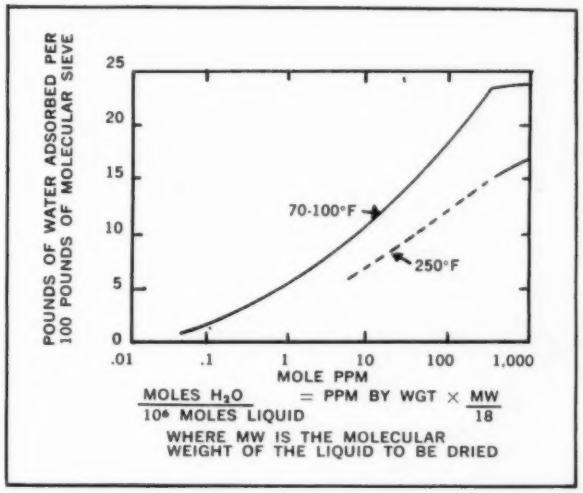

POUNDS OF WATER ADSORBED PER 100 POUNDS OF MOLECULAR SIEVE
MOLE PPM
70-100°F
250°F
MOLES H2O / 10⁶ MOLES LIQUID = PPM BY WGT × MW / 18
WHERE MW IS THE MOLECULAR WEIGHT OF THE LIQUID TO BE DRIED

*Fig. 11. Liquid drying equilibrium data (molecular sieve Type 4A).*

perature, which remains almost constant, is chosen so that desorption readily occurs when a moderate reduction in pressure is made. Because time is not required for heating and cooling the beds, this cycle can be designed to operate more rapidly than the thermal swing cycle.

(3) *Purge Gas Stripping Cycle.* The molecular sieve bed is purged with a non-adsorbable gas, and, since the partial pressure of the adsorbed component in the vapour phase is reduced, desorption will occur.

(4) *Displacement Cycles.* The material may be desorbed from molecular sieves by introducing another adsorbable product which displaces all or part of the previously adsorbed material. A displacement cycle is useful when the material to be desorbed is heat sensitive.

## Industrial Uses

(1) *Drying—Gas Drying.* The advantages of using molecular sieves for gas drying were enumerated and illustrated by Figs. 8, 9 and 10, which demonstrate that molecular sieves are capable of producing very low dew points, have high capacity for water at low relative humidity and retain their capacity at high temperatures.

*Liquid Drying.* Molecular sieves will dry liquids to less than 1 ppm and have a high capacity for water in liquid drying duties. Fig. 11 illustrates the high capacity of molecular sieves for drying non-adsorbed liquids and liquids that are generally classified as being non-miscible with water.

(2) *Purification*

(a) A molecular sieve process was described in which 12% carbon dioxide is removed from exothermic annealing gas.

(b) An installation which removes carbon dioxide from ethylene was described. Two adsorbers, each containing 15,000 lb of molecular sieves, are utilised to reduce the $CO_2$ content of the ethylene stream from between 1000 and 3000 ppm to less than 1 ppm.

(c) A new, commercially proven process to sweeten liquid

hydrocarbons was described. Details of liquid propane sweetening were given in which molecular sieves reduce hydrogen sulphide content to less than 0.02 gr/100 scf.

### Separation

"Union Carbide" molecular sieves Type 5A will adsorb normal paraffins while excluding isomers and cyclic hydrocarbons. As an example, a study was made of an octane improvement process in which a molecular sieve vacuum cycle is incorporated in a 4000 bpsd pentane-hexane isomerisation unit. The economics of isomerisation with molecular sieve separation and isomerisation with fractionation were compared and a daily saving of $668 in favour of the molecular sieve separation was shown to result.

# Adsorption of Solvents in the Liquid Phase of Active Carbon

by A. D. BRYAN*

A. D. BRYAN described the use of active carbon for extracting a solvent from a carrier liquid, which, in most cases, is water, and he discussed some practical applications of the selective adsorptive property of active carbon.

He noted that it is rare, but not unknown, for an aqueous solvent to arise from a process other than that of solvent recovery by active carbon, and he outlined briefly the mode of operation of a solvent recovery plant. Thus, solvent-laden air is directed through a bed of activated carbon which extracts the solvent and passes the stripped air to atmosphere. Live steam, admitted to the charged carbon bed to regenerate it and distil the adsorbed solvent from the carbon, passes as a solvent-rich vapour to a heat exchanger. Usually of the water-cooled surface type, this condenses solvent and water. The condensed water is incidental to the convenient use of live steam as the desorbing agent. If the solvent is immiscible with water, the condensate separates by decantation, so readily offering the solvent for re-use and permitting the water to be discarded. This system is either incomplete or inapplicable if the recovered solvent, or in the case of a mixture, one of the solvents, is soluble or partly soluble in water.

If the recovered solvent is completely soluble in water, it is usual to effect final separation by distillation—sometimes by azeotropic distillation.

Occasionally a more complex solvent mixture may include a component which might not be only partially soluble in water, but also soluble in one of the other components of the solvent mixture. Indeed, such conditions occur, for example, in producing moisture-proofed viscose film known as cellophane, which is proofed by a surface lacquer. One lacquer uses toluene and ethyl acetate as solvents.

Ethyl acetate is approximately 8 per cent soluble in water, depressed to approximately 4 per cent in the presence of a roughly equal volume of toluene. Its recovery in liquid phase from water is widely practised by means of active carbon adsorbent, and this application may be regarded as typical of solvent recovery in the liquid phase.

### Some Selective Adsorption Tests

A number of tests were then described which were carried out to determine the performance of active carbon for the extraction of a number of solvents, from their aqueous solutions. The solvents included ethyl acetate, acetone, methyl ethyl ketone, ethyl alcohol and, finally, carbon disulphide. The data obtained included the break points, the amount of solvent retained per unit mass adsorbent, the quantity of steam required and the effect of solvent concentration.

In the case of carbon disulphide the application arises from the formation of saturated aqueous solutions of carbon disulphide when water is used as a buffer during the bulk loading of carbon disulphide. The tests revealed that active carbon adsorbent recovered the carbon disulphide completely from such a solution with a percentage retention of more than 25 per cent over the first four cycles.

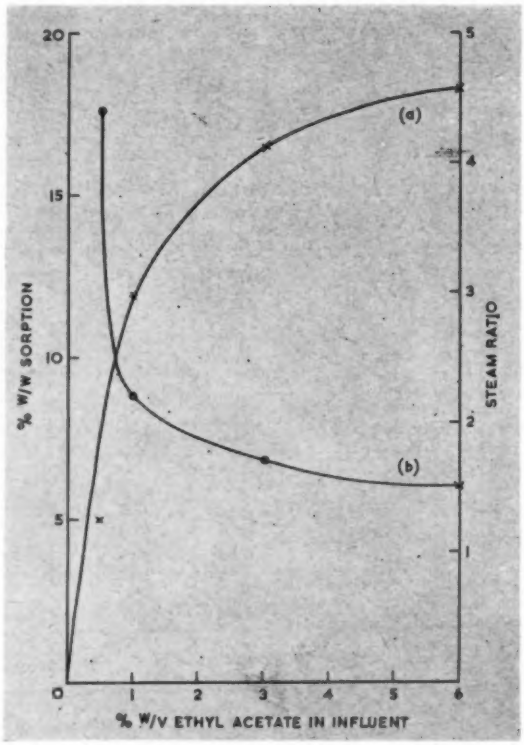



*Fig. 12. An experimental result described by the author (A. D. Bryan). Relationships between ethyl acetate concentration on influent and (a) percentage sorption; (b) steam ratio.*

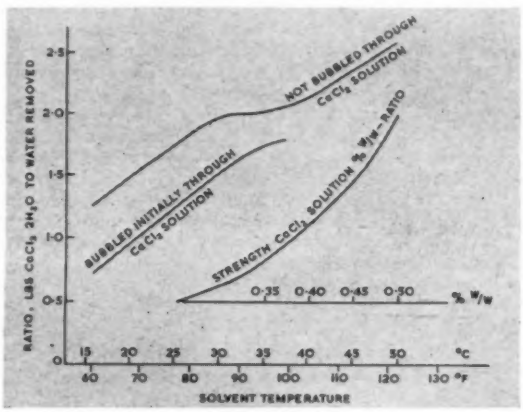

RATIO, LBS $CoCl_2$ $2H_2O$ TO WATER REMOVED
NOT BUBBLED THROUGH $CaCl_2$ SOLUTION
BUBBLED INITIALLY THROUGH $CaCl_2$ SOLUTION
STRENGTH $CaCl_2$ SOLUTION % W/W-RATIO
% W/W
SOLVENT TEMPERATURE

*Fig. 13. Dehydration of 60/40 ethyl acetate/toluene with solid calcium chloride. $CaCl_2 2H_2O$.*

* Technical Engineer, Solvent Recovery Section, Sutcliffe, Speakman & Co. Ltd., Leigh, Lancashire.

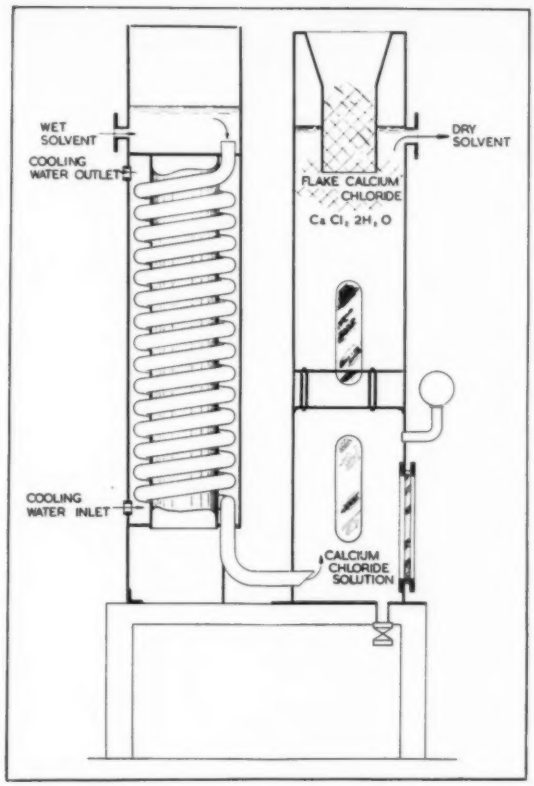



*Fig. 14. A typical dehydrator employing a cooling coil and flake calcium chloride.*

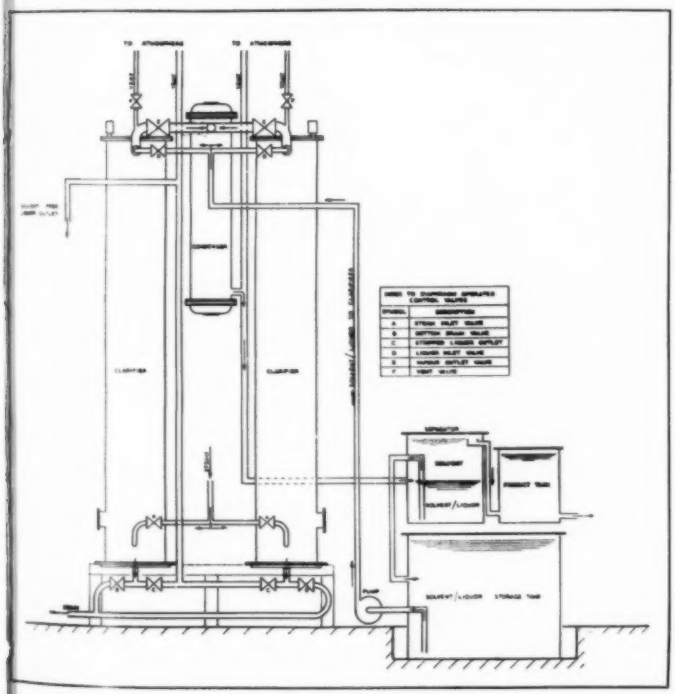


*Fig. 15. Arrangement of automatically-controlled liquid-phase active carbon recovery plant.*

On the plant scale the results indicated that a bed of the same quality of carbon as used in the tests, and having a cross-sectional area of 1 sq ft and a depth of 30 in., would successfully treat over 2000 gallons of contaminated water before the break point was reached. Steaming would be required at approximately 6-hr intervals, and would be used in the ratio of approximately 5:1.

## Commercial Liquid Phase Adsorption Plant

Increasing production of moisture-proofed viscose film quickly offered an opportunity for liquid phase adsorption to deal with the aqueous solutions of butyl and ethyl acetate—the esters in general use. Fig. 15 shows the main features of such a unit. In this plant a riser pipe is fitted to ensure a flooded clarifier, so preventing channelling through the carbon bed. The aqueous liquor, which decants in the separator, is recycled. In many cases, however, separate decantation is not practised, and the distillate from the carbon condenser is fed to the upper layer of the active carbon plant separator. The operation of the valves, shown in Fig. 15, is by mechanical process timer incorporating a wide-range timing selector. This provides for automatic operation of the following cycle:

| | | | | |
|---|---|---|---|---|
| No. 1 | Adsorb | | Drain | Steam |
| No. 2 | Drain | Steam | Adsorb | |

An extensive research into the cyclic efficiency of these units led to the adoption of clarifiers of approximately square cross-section and with a high length to width ratio. The optimum conditions of minimum steam ratio and maximum cycle efficiency were obtained with liquor flow downwards and steam flow upwards, and it was found that the clarifier acted as a packed column refluxing to produce vapour of little less than azeotropic composition.

Practical difficulties which were overcome in the design were the hammer effect, due to waterlogging, and the entry of particles of carbon into the steam distributor. The most satisfactory solution for both problems is a shallow bed of carbonised briquette for supporting the carbon, with a screen interposed to form the distributor.

## Pressure Drop Characteristics of Active Carbon Beds

The following formula represents the pressure loss through a bed:

$$\Delta P = \frac{0.5\,UG}{Sd^2}$$

where $G$ = mass rate of flow, gm/sec/cm$^2$;
$U$ = viscosity of liquid, poises, gm/sec/cm;
$S$ = density of liquid, gm/cm$^3$;
$d$ = mean diameter of particles;
$\Delta P$ = pressure drop across bed, cm w.g./cm bed depth.

The following assumptions are made:

(1) The liquid in question is water at 150°C. Therefore, the values of $U$ and $S$ may be taken as 0.0114 poises and 1 gm/cc respectively.

(2) The value of $d$ is the mean of the screen apertures:

e.g. 8-10 BSS = ... ... ... ... 0.1864 cm

$$\text{10-14 BSS} = \begin{Bmatrix} 50\% & \text{10-12 BSS} \\ 50\% & \text{12-14 BSS} \end{Bmatrix} = 0.1422 \text{ cm}$$

$$\text{14-18 BSS} = \begin{Bmatrix} 50\% & \text{14-16 BSS} \\ 50\% & \text{16-18 BSS} \end{Bmatrix} = 0.1016 \text{ cm}$$

(3) The bed of carbon under consideration is closely packed.

(4) That increase in pressure drop is proportional to increase in bed depth.

## Special Features of Clarifiers

Clarifiers can sometimes preferentially adsorb one of two solvents which may be present in an aqueous solution. An instance is the weak solvent liquor decanted from an active carbon plant recovering ethyl acetate and toluene from moisture-proofing of viscose film in which the alcohol from the wetting of the nitrocellulose is also evaporated to the recovery plant.

The carbon in the clarifiers selectively adsorbs the ethyl acetate and allows the alcohol, which would otherwise build up to undesirable proportions in the recovered solvent mixture, to pass through.

Some solvents have an affinity for water. In cases where the recovered solvent is insufficiently free from moisture, further processing of the solvent or solvent mixture is needed, and in particular cases, for example, the recovery of ethyl acetate and toluene, from moisture-proofing of viscose coated film, dehydration is effected by calcium chloride.

A typical dehydrator (Fig. 14) will employ a cooling coil and thereby economise in calcium chloride (Fig. 13). The moist solvent is first cooled, and then enters the calcium chloride solution flowing upwards, so that the moisture is progressively adsorbed from the solvent by contact with, first, the calcium chloride solution, and then the flake calcium chloride with which the unit is charged.

### A Continuous Carbon Bed

This paper opened with references to adsorption by active carbon of solvents in the vapour phase, and it would seem fitting to close with a brief mention of a recent development in this field.

In concluding his paper, MR. BRYAN described a recent development in vapour phase adsorption equipment, a package-type plant designed around a slowly rotating adsorber. This employs no sequence valves, the segmental bed of the adsorber being arranged to slide through a zone of continuous steaming (Fig. 16).

ACKNOWLEDGMENT

The author expressed his indebtedness to the directors of Sutcliffe, Speakman & Co. Ltd. for their permission to give this paper.

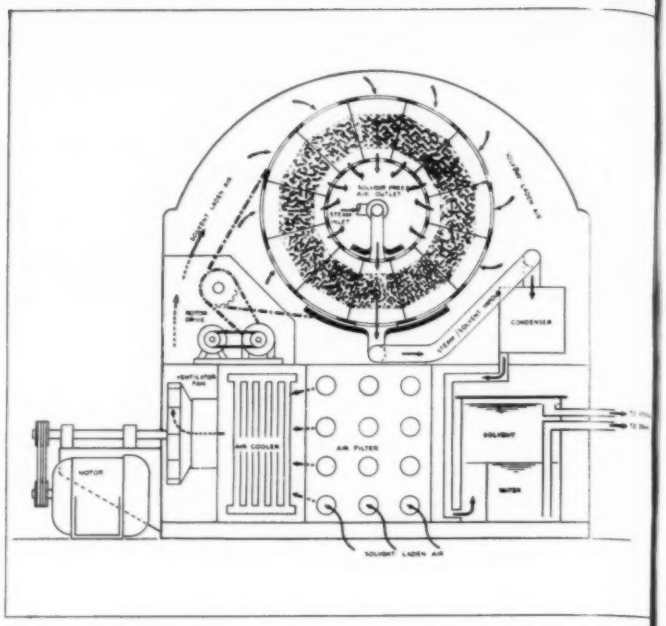



*Fig. 16. A continuous solvent recovery unit employing active carbon.*

# Recovery of Nickel and Chromium in the Electro-plating Industry

A REPORT covering the experiences of 42 firms in the electro-plating industry has, as its main objective, the possibility of reducing losses of nickel, which costs nearly £870 per ton, as well as chromium, and the control of effluents from electro-plating processes.*

Nickel and chromium salts used in these processes are largely wasted by "drag-out" which occurs when plated articles are removed from the plating bath, taking with them solution. Out of an estimated total weight of 2870 tons of nickel used by the plating industry in 1958, about 290 tons represent the metal content of the nickel sulphate and chloride lost from this source. These chemicals increase the total material cost by about 13 per cent, but by the use of simple static rinse tanks it has been found possible to eliminate three-quarters of the loss of these salts. Tanks of this kind are cheap, and in manually-operated plants the outlay may be recovered within a year. It is estimated that the cost of such a tank, complete with pump and pipework, would be in the range £130-£260 according to whether the plant is a small or large one.

Alongside such a tank it would be possible to install an evaporator to concentrate the weak solution, to yield a concentrate suitable for use as make-up in the plating baths. The most important factor in ensuring success of this method is close control of water, since any excess will increase considerably the cost of evaporation.

Chromium recovery appears to be treated by the platers somewhat differently and the "drag-out" recovery tanks are looked upon rather as an insurance against excessive discharge of toxic chemicals than as a means of conserving chromic acid. Chromium-plating differs from nickel-plating in that chemical, or plating-out purification is not practised.

Another feature of chromium-plating is that it evolves large volumes of hydrogen which carry away solution as a fine mist. Of the total loss of chromium, this can account for as much as 20 per cent. This loss is reduced either by floating small sealed-toughened glass tubes upon the surface of the plating solution or by adding a surface-active agent to the solution. Of the two methods, the latter is preferable, although it is regarded by some platers as costly.

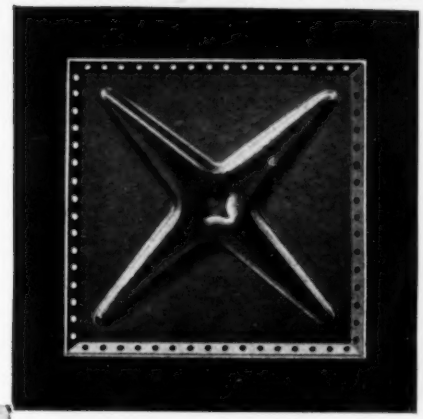
The economic use of water is another aspect of the plating industry considered in this survey; the operation offering the greatest possibility of saving being that of rinsing. Improved methods of rinsing include the use of static "drag-out" tanks, counterflow rinses and ion-exchangers.

Ion exchange is also seen to be a very attractive technical proposition for recovering nickel and chromium and for purifying high-quality water from mixed effluents, and for purifying chromic acid baths. In the field of effluent treatment ion exchange provides an attractive alternative to chemical treatment; it permits the recovery of rinse water so that it can be recycled in an exceptionally pure form.

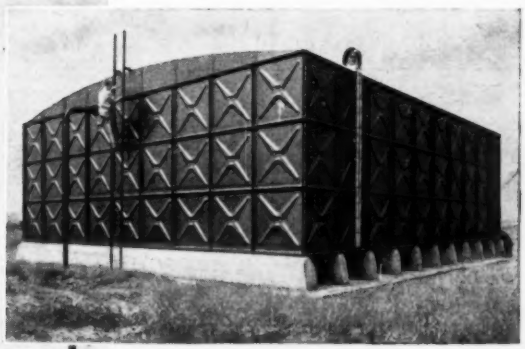
* "A Survey of Nickel and Chromium Recovery in the Electroplating Industry"; E. P. Harris, Department of Scientific and Industrial Research, December 1960.

BCE 9058 for further information

# BRAITHWAITE Pressed Steel Storage Tanks



Effective storage (from 220 gallons upwards) for water, heavy fuel oils and other liquids. These tanks, which can be enlarged or altered to meet varying needs, are light, strong and simple to erect with all tools supplied.



(left) 27,600 gallon covered tank 24′ square × 8′ deep with access ladders, supporting structure and valve control platform, supplied and erected at a housing estate.

(inset) Another covered tank for agricultural estate development, 40′ × 30′ × 12′ deep, capacity nearly 105,000 gallons.

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*A particle classifier in the Chemical Engineering Laboratory of the Bradford Institute of Technology.*

# THE TRAINING OF POWDER TECHNOLOGISTS IN GREAT BRITAIN—AN ALTERNATIVE

by V. C. MARSHALL*

DR. H. E. ROSE'S paper, "The Training of Powder Technologists in Great Britain" (*British Chemical Engineering*, September 1960), is a very important contribution to a field of rapidly growing importance and yet, at the same time, it contains a number of features which, in the writer's opinion, will prove unpalatable to the chemical engineering profession.

The paper is important, because it draws attention to a branch of technology which has tended to lag and also because it makes proposals for teaching the subject as a unified whole. Where the paper will prove unpalatable, the writer believes, is in its attitude to the chemical engineer. For it does not appear to recognise that a chemical engineer is, by virtue of his training, qualified in the technology of processing powdered solids, nor does it recognise that this technology is a branch of chemical engineering. Moreover, in detailing qualifications suitable as a preliminary for candidates about to undertake a specialised training in the subject, chemical engineering qualifications are not even mentioned. Instead, it is proposed, in the main, to train pure scientists with qualifications in subjects such as chemistry, physics and biology.

Now, whilst there is everything to be said for familiarising science graduates about to enter powder processing industries with aspects of powders which are outside their normal studies, the fact remains that, at the moment, the chemical engineer is the best powder technologist that we have. It would appear logical, therefore, to start from the chemical engineer in order to produce a specialist in the field.

The superiority of the chemical engineer is due to the very wide range of techniques involved in the processing of solids in the chemical and allied fields which feature, in addition to the problems of transportation, such items as fluidisation, filtration, catalysis, adsorption, separation of solids from gases, proportionation of reactants, spray drying, mixing, etc. It is not surprising, therefore, that the processing of solids in varying states of subdivision forms a considerable part of chemical engineering training. This can be seen from the *Transactions of the Institution of Chemical Engineers*, which devotes about a quarter of its space to topics such as those listed above. The text-books also devote a great deal of space to solids handling, a standard work such as Brown's *Unit Operations* giving well over a third of its text to the properties and processing of solids. Indeed, the degree of overlap between the detailed course given in the paper and a chemical engineering syllabus for degree or Dip. Tech. must surely exempt schools of chemical engineering from the claim that "(apart from the teaching of mathematics, physics and chemistry) . . . the remainder of the subject has little in common with those normally taught in the applied science departments. . . ."

Though the course proposed overlaps a standard chemical engineering course to a considerable extent, there are some surprising omissions. Though the paper refers several times to the need for the powder technologist to be conversant with heat transfer between solids and gases, this rather specialised topic is not mentioned in the detailed syllabus.

* Chemical Engineering Department, Bradford Institute of Technology.

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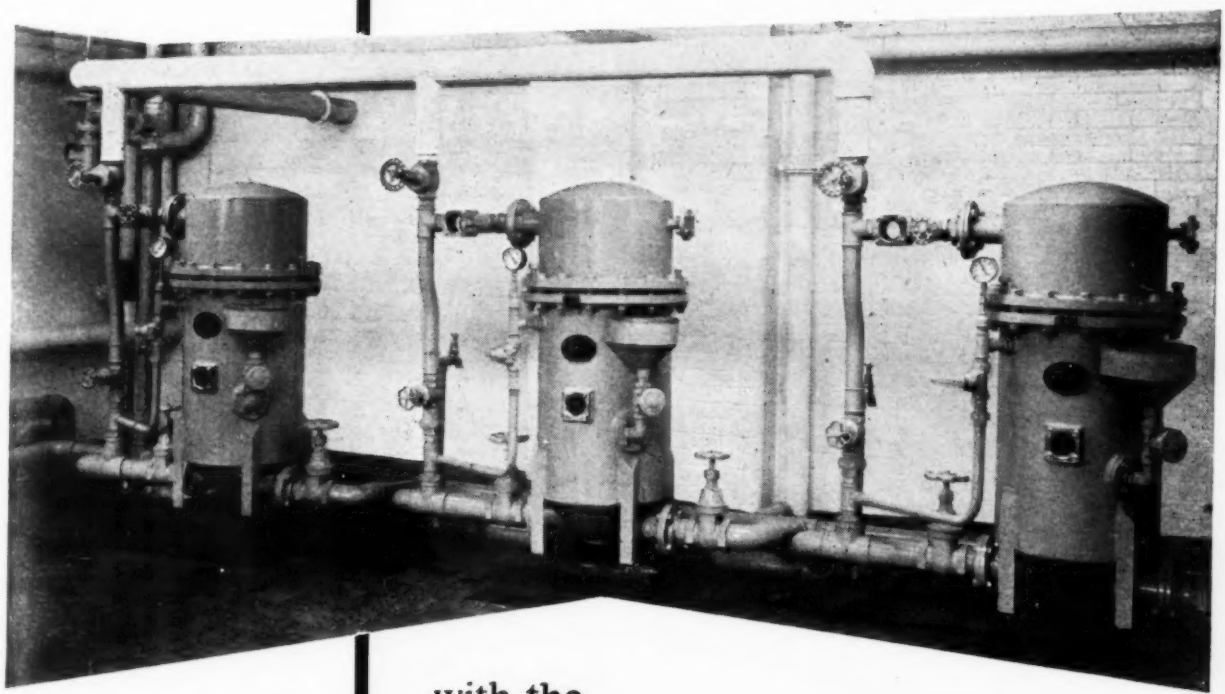

* *They may be: WAXES—OILS ESSENCES—WINES—SPIRITS JUICES—CHEMICALS PHARMACEUTICALS SYNTHETIC RESIN SOLUTIONS SOLVENTS—VARNISHES INDUSTRIAL FLUIDS—and, of course, WATER*

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though it is very unlikely that the background of some of the graduates entering the course will include the necessary concepts. Again, one would have expected to find fluid mechanics as a background subject, in view of the more or less profound influence exerted by fluids in most systems in which particles are handled. Most surprising of all, the course does not introduce any consideration of economics, though improvement in the economy of the powder handling industry is cited as the main reason for having the course. In the writer's opinion it is not wise to assume that the question, of whether or not a particular step will improve a process economically, can be decided by the exercise of common sense alone. For this reason it is highly desirable that all technologists should receive a grounding in elementary economics.

There is, however, a more fundamental reason why the course is not the best answer to the problem and that is that it does not accord with the present trend of thinking on the training of technologists. Instead of training technologists by giving a short course to graduates in pure science, the better approach is to aim at producing a technologist from the very outset, an approach which characterises the courses leading to Diplomas in Technology.

Ideally, then, if industry demand people with the qualifications under discussion, the solution would lie in the establishment of a Dip. Tech. in Powder Technology. Failing this, and without the necessity to establish any new course, the best plan for industries experiencing problems in powder processing is to sponsor industry-based students to take a Dip. Tech. in Chemical Engineering. Such industry-based students could spend part of their industrial training, say two six-month periods, in practical work in powder technology. Industrial training is an integral part of the Dip. Tech. course, ranking as equal in importance with academic study, and is arranged in the closest consultation with the staff of the college who advise on the most suitable type of work consistent with the academic level of the student. From such a course the industrial concern would obtain the services of a qualified chemical engineer with a carefully planned industrial experience including the practice of powder technology.

If industry should feel the need for a greater degree of specialisation, then arrangements would have to be made to run short, advanced courses for Dip. Tech. chemical engineers at one or more Colleges of Advanced Technology which would have to possess the necessary equipment. Such courses could concentrate on the advanced aspects only, as the students would already have a grounding in heat transfer, fluid mechanics and, at the minimum assessment, the elements of the theory and practice of powder technology.

It should not be assumed from the above that there are no grounds for criticising the teaching of the branch of chemical engineering in question. On the contrary there are grounds for believing that it is less well taught than those sections such as heat transfer, mass transfer, etc., which yield more readily to mathematical treatment. But there is another reason. Though powder technology is criticised for empiricism, in fact the opportunities for experiment in chemical engineering laboratories are limited by the size and expense of the equipment involved. Whereas, for example, very useful work both in teaching and research in heat transfer can be performed on a single tube, this kind of approach is seldom satisfactory in experiments with powders where wall effects are important and scale up difficult. The apparatus is often too complicated to make oneself and may be commercially available only in large sizes. With expensive apparatus, intensive use is essential—if it is to be run only one day a year, the cost of that day's run will be fabulous. Those responsible for planning laboratories naturally bear these things in mind and they militate against the provision of apparatus adequate for teaching powder technology as a branch of chemical engineering. There is need for more contact between the schools to exchange experience on the most suitable types of apparatus and how best to use it for teaching purposes.

Though it follows from all this that the arguments in DR. ROSE'S paper in favour of the concentration of facilities for advanced work and research are very soundly based, it is not necessarily true that a university is the best or the only place to possess such facilities. If, without the establishment of special courses, the Dip. Tech. student based on the powder-using industries will prove to be the most satisfactory technologist, then it would be logical to conduct the advanced work in a College of Advanced Technology.

## Reactor Operation Courses at Hawker Siddeley

THE Hawker Siddeley Nuclear Power Co. Ltd., of Sutton Lane, Langley, Bucks., have arranged a basic and an advanced course on the "Jason" reactor to train people in reactor operation.

The *Basic Course* gives the student the opportunity of working at the control console of an operating reactor and taking part in the operational procedures while the reactor is used on its various day-to-day tasks.

The student will be instructed in the routine reactor instrumentation check-out and will be able to operate the reactor controls under supervision during the various stages of reactor start-up including source insertion, moderator insertion, withdrawal of control rods to achieve criticality, controlled increase of reactor power, trimming, controlled power reduction, and reactor scram (which includes water dumping), by various safety trips and interlocks. Other techniques, such as fuel handling, insertion and removal of experiments, routine checks and maintenance, will be carried out in accordance with the reactor programme.

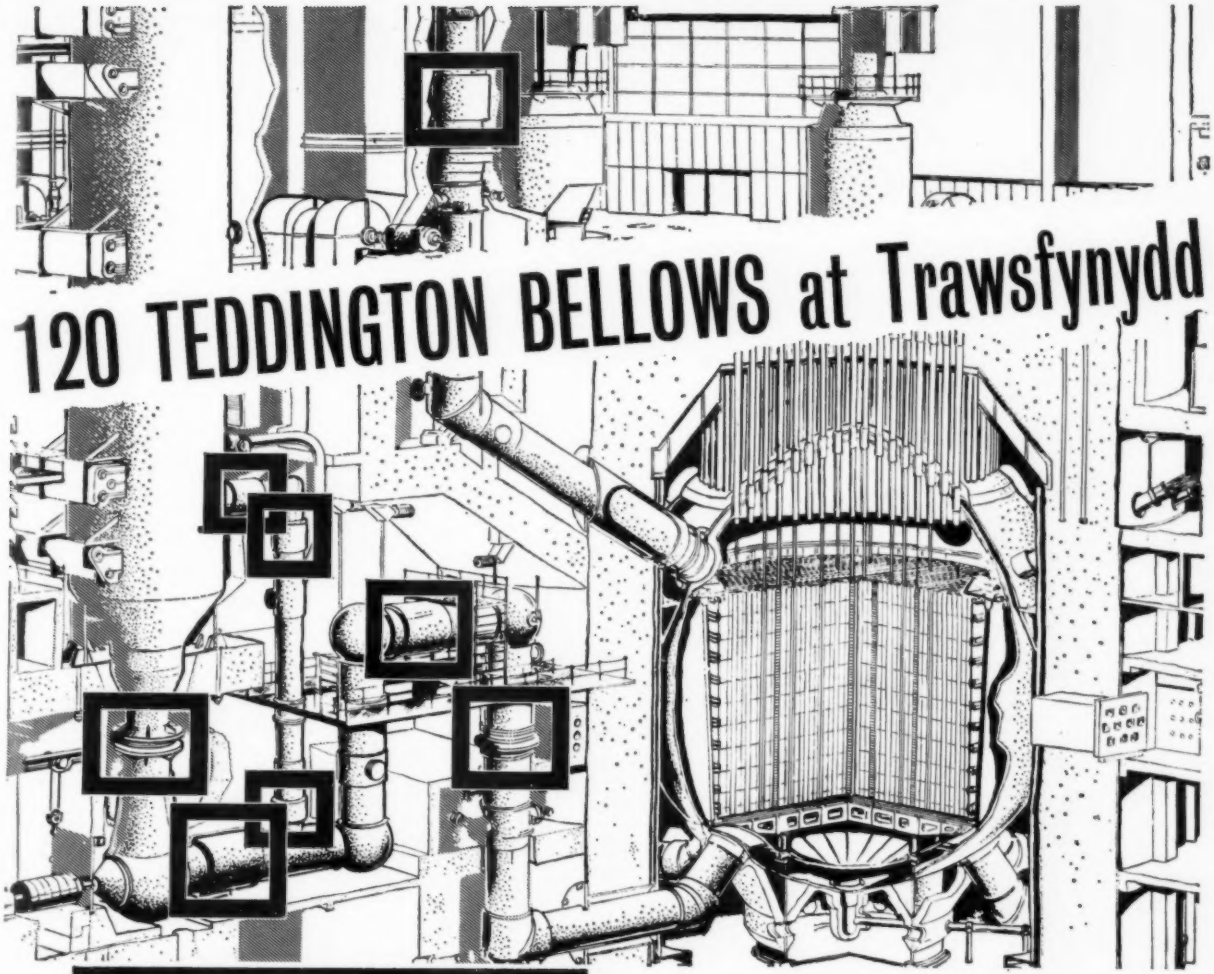
The charge per student for the Basic Course is £5 5s. per day (9 a.m. to 5.30 p.m. Monday-Friday) and the total duration is optional and can be arranged to suit the requirements of the participants.

The *Advanced Course* gives students an opportunity of carrying out particular experiments with the reactor which are of an instructional nature and designed to give the students a closer understanding of the reactor's characteristics. The students will also be able to operate the reactor controls under supervision and take part in routine maintenance checks. For a fair proportion of the course the reactor will be available for the exclusive use of the students. Such experiments may also depend upon the availability of equipment, as well as the approval of the Reactor Safety Committee.

The charge per student for the Advanced Course is £15 15s. per day (9 a.m. to 5.30 p.m. Monday-Friday) and again the duration is optional, but a period of 3 to 5 weeks is recommended if a thorough working knowledge of the reactor is to be obtained.

All inquiries and applications for attendance at the courses should be addressed to MR. B. D. BAINES, Technical Sales Manager, Hawker Siddeley Nuclear Power Co. Ltd., Sutton Lane, Langley, near Slough, Bucks., England.

# 120 TEDDINGTON BELLOWS at Trawsfynydd



*By courtesy of 'NUCLEAR POWER'*

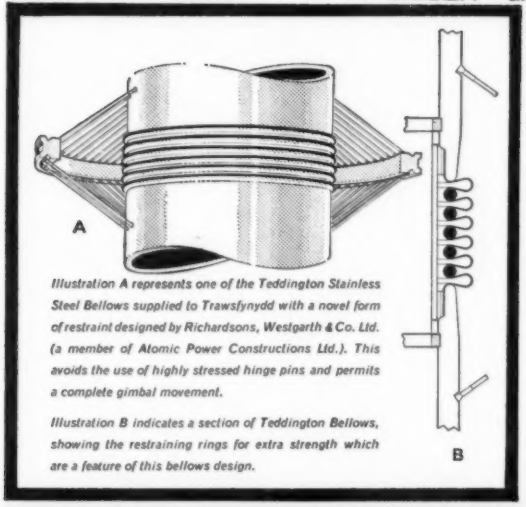



*Illustration A represents one of the Teddington Stainless Steel Bellows supplied to Trawsfynydd with a novel form of restraint designed by Richardsons, Westgarth & Co. Ltd. (a member of Atomic Power Constructions Ltd.). This avoids the use of highly stressed hinge pins and permits a complete gimbal movement.*

*Illustration B indicates a section of Teddington Bellows, showing the restraining rings for extra strength which are a feature of this bellows design.*

**NO LESS THAN 84 BELLOWS 66″ diameter and 36 BELLOWS 42″ diameter, all of butt-welded stainless steel manufacture, are being fitted at Trawsfynydd to take up movement in the $CO_2$ cooling gas ducts.**

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AMERICAN LETTER

# United States Progress in Saline Water Conversion

A GREAT deal of work has been accomplished and some important discoveries made in the field of saline water conversion since Congress passed the Saline Water Act of 1952. This Act, which at first provided for an expenditure of $2 million for development purposes, was followed by an Amendment increasing the appropriation to $10 million and a later Resolution authorising the construction of at least five demonstration plants for a further $10 million in order to establish the most favourable methods.

## The Current Federal Programme

After close collaboration between the Department of the Interior, eminent scientific authorities and leading industrial concerns, a decision was reached to build the following types of plant:

- A long-tube, multiple-effect evaporation unit, designed to operate on sea water and having a capacity of one million gallons per day.
- A multi-stage flash distillation unit, operating on sea water and also having a daily output of 1 million gallons.
- A vapour-compression, forced-circulation plant, operating on brackish water and having a capacity of 250,000 gallons per day.
- An electro-dialysis unit with a daily output of 250,000 gallons when operating on brackish water.

The first type of plant will be built to the designs of W. L. BADGER and Associates of Ann Arbor, Michigan, and will be installed adjacent to the Freeport, Texas, plant of the Dow Chemical Co., which will supply the steam and use the brine and fresh water produced. As a measure of their confidence in vertical, long tube, multiple-effect evaporation, the designers have recently formed the Seawater Conversion Corporation in conjunction with another United States company and will offer these plants for sale throughout the world.

Several types of multi-stage flash distillation units were considered and a proposal to the designs of the Fluor Corporation for a unit of this type, based on optimisation by a computer, was selected. This plant will operate in conjunction with a nuclear reactor, and once a site favourable to public safety, nuclear waste disposal and an economic use of the water produced has been found, construction is expected to proceed rapidly. These studies indicated that a similar larger unit containing 52 flash chambers ought to produce 50 million gallons per day at a cost of 42 cents per 1000 gallons.

Vapour-compression, forced-circulation techniques have been developed to a high degree by such firms as Griscom-Russell and Cleaver-Brooks, who are among several contenders for a plant in this category to be installed in Roswell, New Mexico. As regards electrodialysis, it has been decided to install such a unit in Webster, South Dakota. An outstanding pioneer in this specialist field is Ionics Inc., of Cambridge, Mass., who are expected to be chosen as contractors for the Webster installation, on account of their accumulated experience in building numerous similar commercial installations in various parts of the world.

The fifth type of plant to be built will use a freeze-separation process and will probably be located on the east coast. Rapid strides have been made in freezing techniques for separating salt from sea water and several commercial methods have been recently announced, any one of which may be chosen by the Government for full-scale demonstration. These include the Blaw-Knox flash-freezing process which is based on the chilling produced by flashing an immiscible hydrocarbon, the Fairbanks-Morse vacuum vaporisation process and the flash-freeze method developed by the Carrier Corporation. A more recent entrant to this field is the Struthers Wells-Scientific Design process which employs the controlled crystal growth of ice in conjunction with evaporation and cooling induced by flashing a hydrocarbon in contact with the sea water.

## Some Limiting Factors

The recovery of potable water from sea water has, of course, been undertaken for many years in large marine units built by such well-known firms as J. & G. Weir, Griscom-Russell, Cochran and Maxim-Emhart. In more recent times, evaporation techniques employed at sea have been improved and adapted to land-based use as, for example, in the huge multi-million-gallon-per-day plants built by Weir and the 28-stage Cleaver-Brooks long-tube flash evaporator to be supplied to the Virgin Islands.

As regards electrodialysis units, such as made by Ionics Inc., an important factor adversely influencing the purity of the product as well as operating costs is a high concentration of salt in the feed water. Hence these units are much more suitable for treating brackish waters than sea water at their present stage of development. Numerous installations ranging from 1000 to approximately 100,000 gpd are now operating in many parts of the world, on brackish feeds containing some 2000 to 5000 ppm of dissolved solids, and are producing potable waters containing about one-tenth of these quantities of undesirable salts. The first United States commercial Ionics unit was recently installed in Coalinga, a small Californian town. This 30,000-gpd unit produces a potable water containing some 500 ppm of dissolved solids from a brackish source containing 2000 ppm for a cost of $1.5 per 1000 gallons, which compares very favourably with $7 per 1000 gallons for potable water brought in by truck.

## Future Developments

The Office of Saline Water discovered that the absolute minimum energy needed to recover 1000 gallons of fresh water from sea water is 2.8 kWh, regardless of the method used, while on a practical scale, the minimum energy requirement is likely to be about four times this figure.

Among the methods now under trial are solar evaporation using large areas of inflated plastic sheeting, rotary stills, wiped film evaporation and hydrate processes using halogenated methanes or similar compounds in alternate cycles of formation and decomposition. Other investigations include the development of advanced osmotic and membrane techniques, the use of chelates, solvent extraction (e.g., triethylamine) and gas hydrate formation via propane.

Several highly imaginative suggestions have also received consideration or initial investigation, such as the combined use of fuel cells and electrolysis, selective precipitation of salts by radiation and the employment of biological means such as algæ or ocean birds. Further stimulation in the field of United States salt water conversion has recently been announced in the form of yet another Senate Bill to authorise an additional $20 million for development work.

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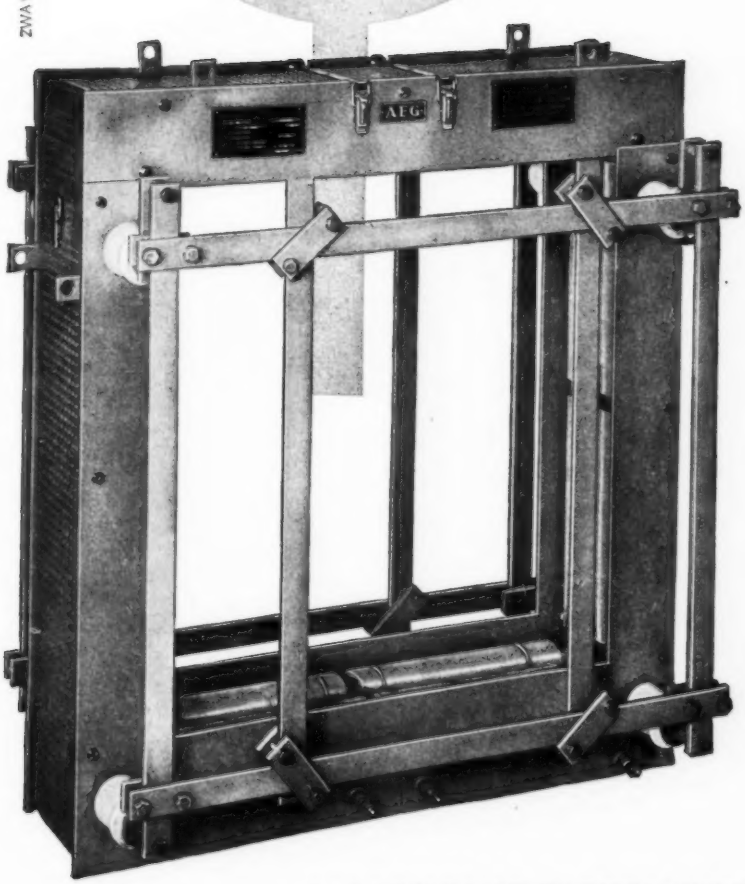


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# VISCOSITIES OF AQUEOUS ETHANOL SOLUTIONS

by D. S. DAVIS

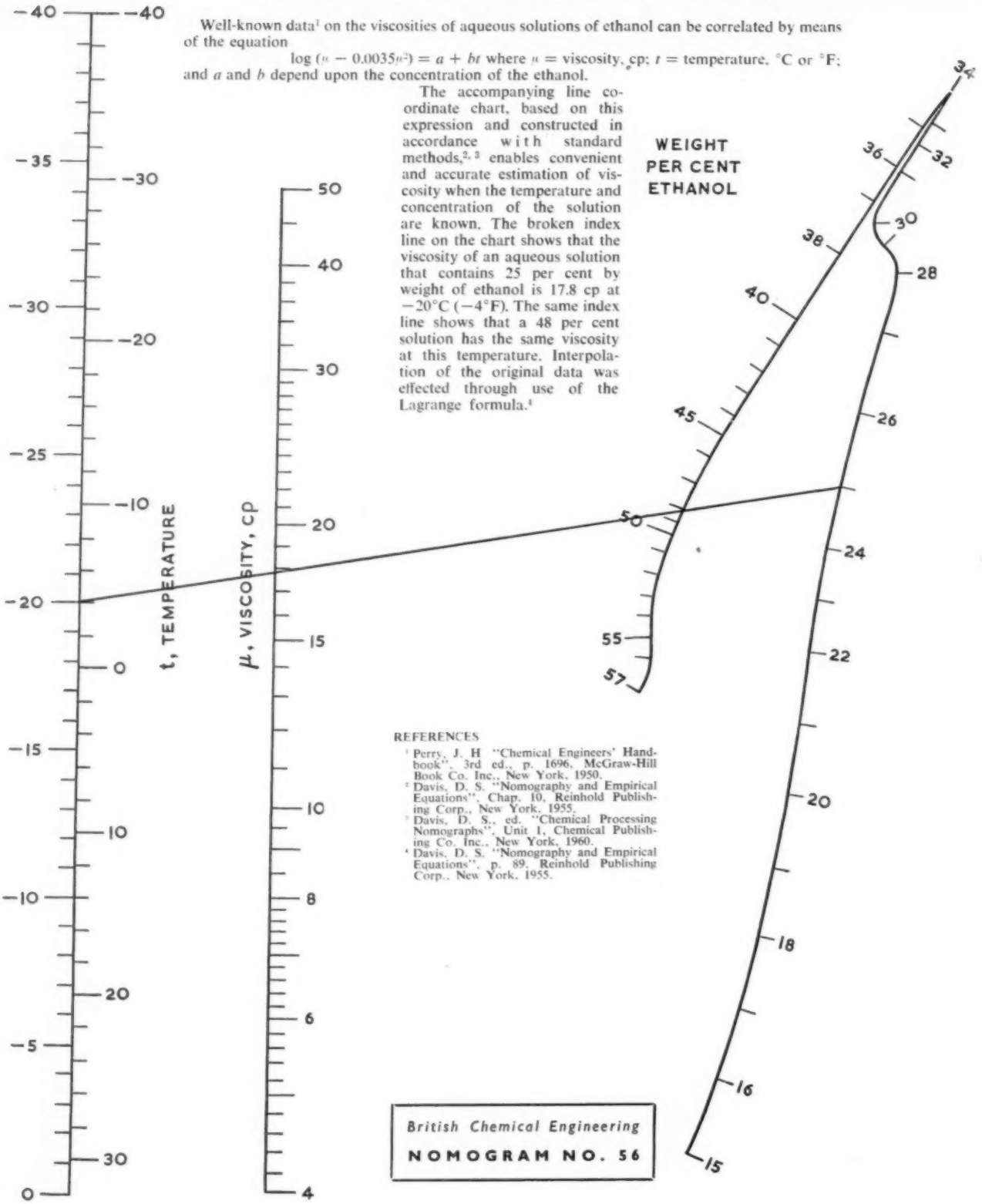
(Head, Department of Paper and Pulp Technology, University of Alabama)

Well-known data[1] on the viscosities of aqueous solutions of ethanol can be correlated by means of the equation

$$\log (\mu - 0.0035\mu^2) = a + bt$$

where $\mu$ = viscosity, cp; $t$ = temperature, °C or °F; and $a$ and $b$ depend upon the concentration of the ethanol.

The accompanying line coordinate chart, based on this expression and constructed in accordance with standard methods,[2, 3] enables convenient and accurate estimation of viscosity when the temperature and concentration of the solution are known. The broken index line on the chart shows that the viscosity of an aqueous solution that contains 25 per cent by weight of ethanol is 17.8 cp at −20°C (−4°F). The same index line shows that a 48 per cent solution has the same viscosity at this temperature. Interpolation of the original data was effected through use of the Lagrange formula.[4]

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[1] Perry, J. H. "Chemical Engineers' Handbook", 3rd ed., p. 1696, McGraw-Hill Book Co. Inc., New York, 1950.
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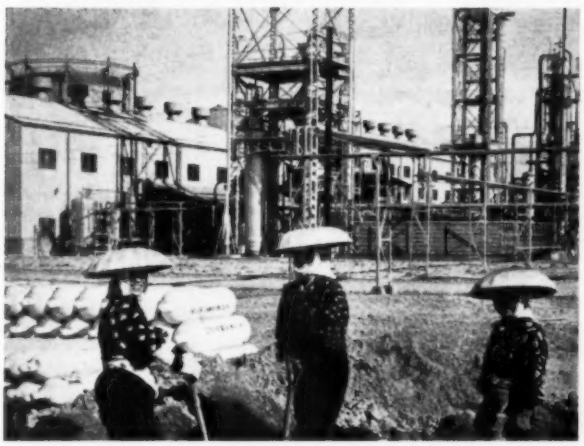

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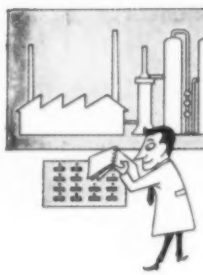


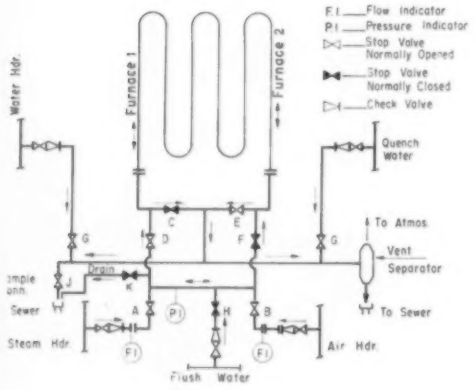
# PROCESS ENGINEER'S NOTEBOOK

## Loosening Coke from Furnace Tubes

THE PROCESS OF DECOKING refinery furnaces consists of cracking and shrinking the coke loose by heating the furnace tubes from the outside while a stream of steam expels the loosened deposit from the interior of the tubes. Although this is a simple enough operation, the pipework manifolds for supplying the services to carry it out are often unnecessarily complicated. This short-coming is eliminated in the system described below.

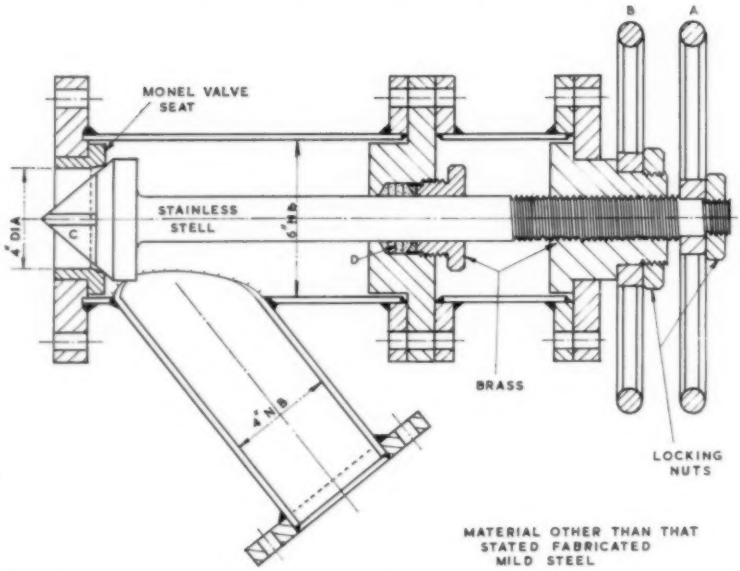
This comprises two interconnected manifolds, one for the supply of steam, air and flush water and the other for effluent, quench water and sampling connection. In the scheme illustrated the two valves marked C and F are closed when coil 1 is to be decoked and are opened when coil 2 is to be treated. Valves D and E are then closed.

Features of this arrangement include dual functions for the valves, a vent separator for the removal of non-condensable gases from the effluent before it is discharged to sewer, and break flanges in order to check for erosion.




(Abstracted from *Pet. Ref.*, 1960, **39**, 4, 218.)

## Self-seating Plug Valve for Classical Fusion Operation




IN THE PREPARATION of synthetic phenol by the classical sulphonation and fusion process, the fusion is carried out in cast-iron fusion pots. These pots have vertical sides with bottoms dished upwards to allow for expansion and to permit complete evacuation through a side nozzle and a bottom run-off valve. When the fusion operation is completed the molten mass is drained through this side nozzle and outlet valve into the quench pots.

Considerable difficulty is normally encountered with this valve due to material freezing in the valve and to difficulties in seating the plug, but a simple fabricated valve made up in accordance with the attached sketch has been found to overcome these problems.

The special plug valve head acts in the manner of an auger bit to drill a passage way through any solidified material in the valve body, and by simultaneous operation of the two hand wheels (A and B) provides a new tight valve seat face for each operation of the valve. The packed gland at D, packed with graphited asbestos because of operating temperature, protects the thread on the valve spindle, and to prevent seizing of stainless-steel surfaces the stainless valve spindle and plug is only in contact with either Monel or brass surfaces.

F. MOLYNEUX

## Photographic Aids

ONE REASON FOR the growing use of photographs as a means of communication is that they provide a very accurate and reliable picture of a physical situation. They are particularly useful where it is no longer practicable to transmit instructions either verbally or by means of rough sketches.

Photographs can accompany detailed instructions for work to be done. Thus, for a particular piece of equipment, its photograph, with its various components numbered and showing its relation to adjacent pieces of equipment, can accompany the instructions outlining how a particular job is to be performed. If a considerable amount of dismantling is necessary, photographs of a previous dismantling of the same equipment will show how the work is to be carried out.

A major repair of process equipment usually provides an opportunity to inspect equipment that is not normally accessible when in operation. Pictures

taken before such work is undertaken establish the condition of the equipment and show the effect of wear and corrosion upon it. Such pictures, apart from illustrating weak points, and possibilities for improved mechanical design, can be used to justify future maintenance or replacement.

Where equipment is liable to failure through corrosion, the record of the appearance of the vessel surface, such as pitting, or blisters in the case of linings, helps to determine the potential life of the equipment and to select a more suitable replacement if its material of construction is unsatisfactory.

Photographs can be very useful at many stages of a new plant. They can help the designer and draughtsman to prepare plant layouts and building modifications. In replanning existing plants, especially where headroom is limited, they can also assist the redesign of a plant and short-circuit much of the drawing work.

Photographs are also helpful for recording revisions or deviations from drawings. These changes can be incorporated into revised drawings at a later stage of the work on the project.

When it comes to preparing maintenance schedules, the nature of the work can be illustrated very easily by photographs and the precise points which require attention and inspection can be marked upon them. These pictures can also be useful to operating staff to illustrate operating procedures and safety precautions.

(Prepared from a paper, "Photographs Speed Field Repair Information", by C. B. HAMMOND, A.S.M.E. Conference, September 1960.)

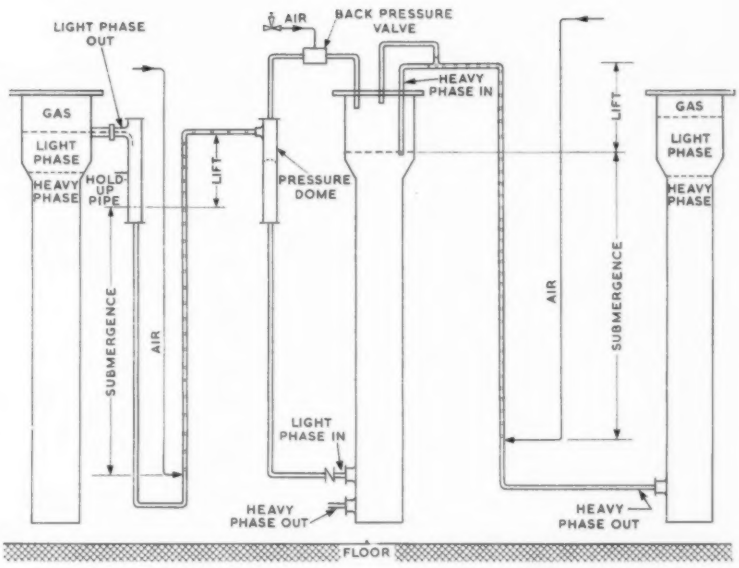
# Liquid-liquid Extraction Air Lift Pump

IN THE PRELIMINARY design of air lift pumps, it is worth bearing in mind that the maximum liquid throughput increases almost directly as the cross-sectional area of the pumping column, provided that the submergence is constant.

By submergence is meant that portion of the total length of any air lift from the point of air injection near the bottom of the pumping column to the static liquid level and it is normally expressed as a percentage of the total pumping column length.

According to a recent publication,* when the pumping column diameter reaches 2 in. the maximum liquid flow rate is three-quarters of that expected when applying the area ratio increase over a column of $1\frac{1}{2}$ in. diameter.

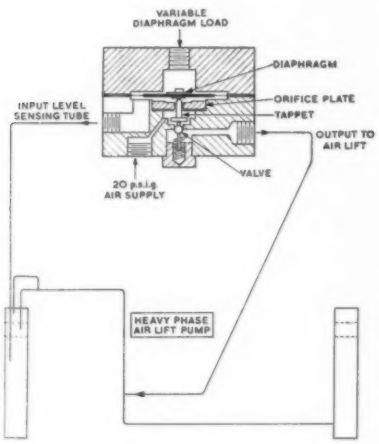
Typical uses for these pumps are shown diagrammatically; the left-hand diagram illustrates a heavy phase pump installation. Worth noting is the air separation provision at the top of the pump which consists of an expanded section in the horizontal run of eductor, carrying a side connection for a vent leading to the top of the same column which the pump is feeding. This addition avoids the mixing of materials of different concentration. Thus, the process columns are made to serve as their own vent system, which is possible when the pressure at the gas-liquid interface is atmospheric.




The other half of this system is shown in the right-hand diagram and refers to the transference of the lighter phase to the main extraction column. As a rule, in an installation of this kind, the pumping column can be made high enough to cause the liquid to flow by gravity through the injection line to the base of the column. Where headroom is limited at the column head, pressure must be applied to the liquid in the injection line to force it into the column. This is carried out by means of a pressure dome in the pipe connecting the pumping column and the injection line. Thus, when air and liquid separate in this dome, air pressure builds up against the back pressure valve in the vent line. The pressure setting of this valve needs to be just sufficient to cause the light phase to enter the column base; usually, a head of a few feet of fluid will meet the case.

Control of the air lift pump for inter-column pumping is very simple because the air lift pump is self-regulating. The air supply to each pump requires a simple flow meter, and level control of the heavy phase is obtained by means of the modified flow controller shown in the third diagram. The level in the column can be adjusted by varying the diaphragm load of the controller; the amount of air fed to the air lift is controlled by a force balancing of the controller diaphragm and the liquid flow rate is controlled by regulating the air flow to the pump.

* "Air Lift Pumps—Characteristics and Application to Liquid-liquid Extraction Systems", J. S. Hurst, Union Carbide Nuclear Co. Report No. Y-1304.



# Book Reviews

### Chemical Analysis
by H. A. Laitinen
McGraw-Hill, New York, 1960, 611 pp., 97s.

THE reasons for a chemical engineer's interest in a book on chemical analysis may be twofold: first, the supervision of sampling and analysis is vital to the control of processes, and secondly, the principles applied by the analyst in the laboratory can also be applied to some types of batch operations to obtain optimum yields for, say, a precipitation process.

LAITINEN'S new book is relevant to both these interests. It is devoted not so much to giving specific experimental methods and techniques of analysis as to a general discussion of the fundamental principles involved in analytical operations. Groups of chapters deal with acid-base equilibria in both aqueous and non-aqueous solutions; precipitates, their formation, solubility, ageing, contamination and colloidal properties; multistage separation processes and electrolytic separations. A brief but adequate section, with a number of worked examples, introduces statistics, and one of its major applications for plant engineers, the theoretical basis of sampling. These are some of the highlights of the volume for chemical engineers, which are not always adequately dealt with in books more specifically on chemical engineering processes. The book also discusses very fully the more usual chemical tools of the analyst, such as oxidation-reduction titrations and other classical wet way methods. Numerical problems (with answers) follow many of the chapters.

This, one of the first volumes in the new McGraw-Hill series in advanced chemistry, is very attractively presented, with only one minor flaw, the absence of an author index to the numerous references given as footnotes on each page. It is a book to be recommended not only to analysts as a general reference text, but also to chemical engineers who are concerned with the control of inorganic processes.

W. STRAUSS

### Translation from Russian for Scientists
by C. R. Buxton and H. Sheldon Jackson
Blackie & Son Ltd., Glasgow, 1960, 299 pp., 30s.

THIS is an invaluable book for the would-be reader of Russian technical literature. It includes a section on grammar and word-formation, translation exercises and a large number of annotated and non-annotated texts on a variety of subjects under the two main headings of physics and electrical engineering, chemistry and chemical engineering. A vocabulary is included with some common abbreviations. It is an excellent work with a very neat arrangement of the contents and very few typographical errors. The only weak point may be the rather small print used for the Russian texts.

M.C.C.

### Principles of Analog Computation
by George W. Smith and Roger C. Wood
McGraw-Hill, New York, 1959, 240 pp.

AS the spelling of the title indicates, this is an American book. It is of interest to chemical engineers, as the main subject is simulation by analogue methods.

The necessity of obtaining a more detailed insight into processes of a chemical or physical nature, say into the manufacture of synthetic fibres or of distillation, and the very great difficulty in using the plant for experimental purposes, calls for equipment which simulates the process. Analogue computers do not compute in the strict sense of the word; they display on a graph or cathode ray tube the effect of varying parameters and conditions—if needed on an accelerated time scale. Their main purpose is to supply some evidence of the correctness or otherwise of a proposed system or of permissible departure from an existing system.

The authors describe first the major components used in analogue computers, the problems of planning and programming, and end with a general theory of the operation of computer components. The most useful portion is in between, namely the simulation of linear processes, a very substantial and useful chapter being devoted to graphs interpreting transfer functions. The examples refer mostly to control problems. The simulation of non-linear systems is discussed by reference to diodes, relays, and other means of generating arbitrary functions.

Each chapter has six to twelve examples and about twenty schematic diagrams.

It would be useful if literature other than American was more adequately quoted and definitions given of terms which are different in American usage from the rest of English literature, otherwise the book is well balanced and illustrated, and should be useful to those who are interested in the deeper understanding of either analogue computers or their application to industrial and other processes.

L. A. STEINER

### Chemistry and Chemical Engineering
by A. J. Bune
The Sunday Times Career Books, London, 1960, 104 pp., 3s. 6d.

THIS book is designed to inform the scholar, and those adults concerned with advising him, more about chemistry and its scope, and the qualifications required for the chemical professions. It should be most useful for science students in school sixth forms who wish to plan their further education and who have not yet made up their minds in which branch of science to specialise. Conditions of employment, pay, prospects and training required are all dealt with and a glimpse of the daily work of chemists is given. There are chapters dealing with research, manufacture of chemicals, petroleum, plastics, food and with the other main branches of the chemical industry. A valuable section at the end of the book deals with some prospective employers, and a special chapter on the part played by chemistry in nuclear science is especially useful.

### Gas Chromatography Abstracts, 1959
Edited by C. E. H. Knapman
Butterworths Scientific Publications, London, 1960, 164 pp., 42s.

THIS book is the second of its type to be published, and records over 700 abstracts of papers on gas chromatography, the majority of which were published in 1959. All the various aspects of gas chromatography have been included, the abstracts being effectively cross-referenced under author and subject indices.

Although many in the gas chromatography field will be grateful to the Editorial Committee of the Gas Chromatography Discussion Group and the 28 abstractors for compiling this comprehensive bibliography, unfortunately, by publication annually in book form, it does not obviate the necessity of scanning the literature for current developments in this rapidly expanding field. A quarterly review on the subject in one of the scientific journals would partially overcome this difficulty and is offered as a suggestion to the Editorial Committee as an alternative or as a supplement to the present arrangement.

P.E.B.

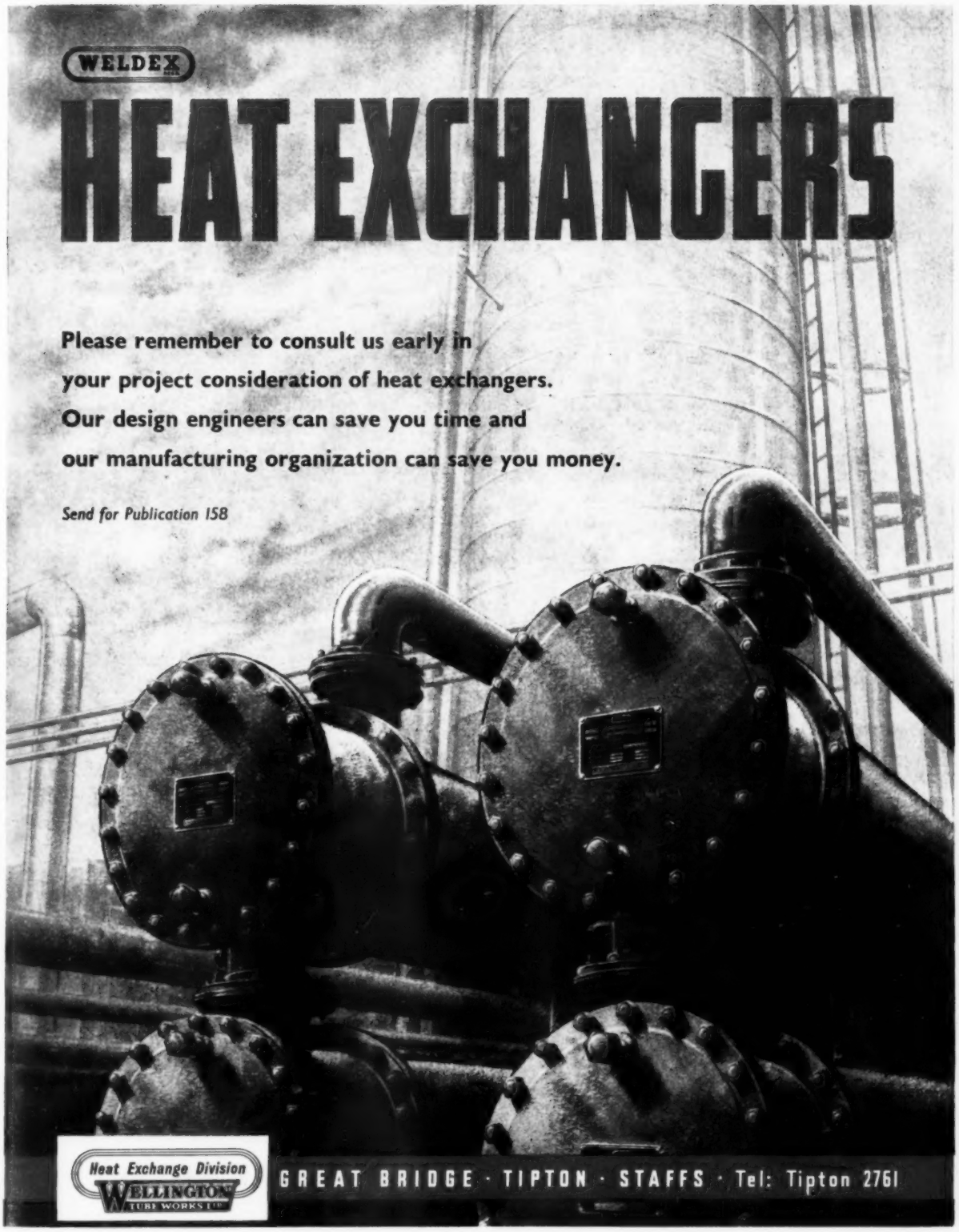
### Rapid Statistical Calculations
by M. H. Quenouille
Charles Griffin & Co. Ltd., London, 1959, 92 pp., 10s.

THOSE of us who learned some statistical methods either during our undergraduate courses or later frequently find that we are at a loss for the correct formula to calculate, say, the mean and standard deviation of a number of values, or a linear regression analysis. This small book presents many of the statistical formulæ used in the correlation of data, together with worked examples. In addition, a number of quick, graphical methods for estimating linear regression, the monotonic association between two time series, etc., are given. Abbreviated tables of functions including Student's t, chi-squared and F-distributions, significance levels, are listed in appendices. This is a most useful booklet at a reasonable price.

W. STRAUSS

### Some Recent Activities of IAEA Division of Public Information. International Atomic Energy Agency, Vienna 1, Austria.

ISSUED PRIMARILY as a service to the specialised Press, this review of activities is likely to be useful to anyone interested in applications of radiation. The booklet briefly summarises the papers presented at a series of conferences including those on controlled thermonuclear fusion, disposal of radioactive wastes, large radiation sources in industry, use of radioisotopes in the physical sciences and industry, and the chemical effects of nuclear transformations.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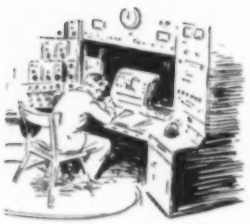
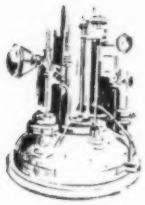


# LETTERS TO THE EDITOR



## Perforated Plate Distillation Columns

**Dear Sir,**

We read with interest the article by B. Melichar appearing in your October, 1960, issue on perforated plate columns for distillation.

Your readers will be interested to know that the designs shown on page 726 of that issue, in Figs. 4, 5, 6, 7 and 8, did, in fact, originate from one of our engineers (indeed, the undersigned).

The design shown in those figures has been covered by the following patents which are assigned to L'Air Liquide S.A.

French: No. 1,074,862
German: No. 950,190
Dutch: No. 78,341

and the diagrams themselves are based upon the French specification.

Yours faithfully,
J. AUCHER

L'Air Liquide S.A.,
Departement Chimique,
75 Quai D'Orsay,
Paris (7e).

## Discharge of Liquefied Petroleum Gases through Orifices*—Author's Reply

**Dear Sir,**

The articles mentioned by Messrs. GOSNEY and EMERSON are of great interest to the present authors; in particular those dealing with non-aqueous liquids. They confirm the well-known fact that a far too low discharge rate is calculated if thermal equilibrium between vapour and liquid at all pressures is assumed during passage through the orifice. This holds also with fluids containing some vapour before entering the orifice.

Better agreement with experience is obtained when the discharge rate is calculated as for an incompressible fluid. The authors agree on this point with the commenters. An empirical reduction factor must, however, be applied with common orifices. So, for instance, with boiling water containing no free steam and applying a contraction factor as for an incompressible liquid a reduction factor of 0.7 to 0.8 was found. Materially smaller reduction factors apply if some steam is present. This behaviour proves that some evaporation takes place, influencing the discharge rate.

KITTEREDGE and DOUGHERTY's formula was mentioned because satisfactory results were obtained in practice using data from their article. VAN WIJK and WESSELING also systematically used the equation of incompressible flow. Following a similar procedure as KITTEREDGE and DOUGHERTY, they did not apply an empirical reduction factor. The amount of vapour liberated was calculated from the condition of maximum mass flow rate. Vaporisation is then considerably less than with equilibrium flow and the corresponding temperature drop is smaller.

Taking, for example, propane at 20°C and 8 kg $cm^{-2}$ (Table IA in the article commented upon), maximum mass flow rate is calculated for a quantity of vapour such that the liquid temperature had fallen to 12.5°C which corresponds to 6.4 kg $cm^{-2}$. The boiling temperature of propane at the downstream pressure (1 atm) is −42°C.

It has been said in the article that some large vapour bubbles were observed moving towards the orifice. In this case and also in the discharge of water containing some steam the finite growth rate of vapour bubbles will probably be the reason, or one of the reasons, why vapour generation is limited.

PLESSET, ZWICK and DERGARABEDIAN studied bubble growth in superheated water. They found that after an initial period the radius of a vapour bubble increases as the square root of the time, $\sqrt{t}$. BRUIJN and VAN STRALEN and the writer found a similar behaviour in mixtures of water and some organic compounds. With water the coefficient of $\sqrt{t}$ is proportional to the degree of superheating. In DERGARABEDIAN'S experiments it took a millisecond for a bubble to grow from a very small radius to 0.1 mm. In these experiments the water was at rest and the results cannot be quantitatively applied to discharge through orifices.

Experiment on bubble growth in flowing fluids will be necessary for a quantitative understanding of the vaporisation during the passage of an orifice. Until this has been obtained, all methods of calculation discharge of boiling liquids through orifices under simultaneous vaporisation are only justified by the degree of agreement with experimental data and may, therefore, be called empirical.

Yours faithfully,
W. R. VAN WIJK

Laboratory of Physics and Meteorology,
Wageningin, Holland.

* *Brit. Chem. Eng.*, 1960, **5**, 252.

## A.B.C.M. Annual Dinner, 1961

**Dear Sir,**

Some of your readers will have experienced an overlapping of the above event with other events in the chemical and allied industries, and with a view to avoiding this in the future, we are asking you to give publicity to the date of the next A.B.C.M. Annual Dinner.

This will be held on Wednesday, October 11, 1961, 7 for 7.30 p.m. (i.e., the evening before the Annual General Meeting of the Association). The venue will be the Great Room, Grosvenor House, Park Lane, London, W.1.

Yours faithfully,
JOAN CONSITT

Intelligence Officer,
The Association of British Chemical Manufacturers.

## Problems of Communication in Science and Industry

*Received from the Press Office of the D.S.I.R.*

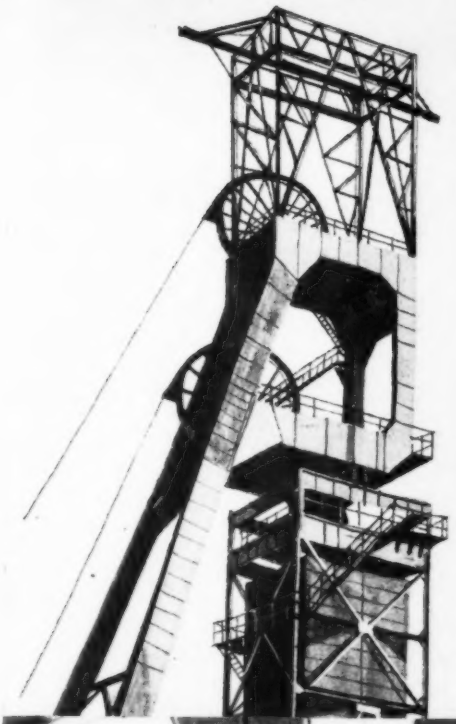
Rapid communication between science and industry is a vital link in modern technology. And today, when over £300 million are spent each year on industrial research and development, effective communication is a problem facing industrial and research management.

Your readers will appreciate the need for research results to be known quickly and clearly to the widest possible technical audience if existing knowledge is to be used with benefit, and for the time lags between scientific developments and industrial application to be reduced significantly.

This, indeed, is to be the theme of a two-day national conference—*Science and Industry—The Problem of Communication*—to be held at the Brangwyn Hall, Swansea, on April 18 and 19, which will deal thoroughly with the many problems of disseminating research results. It is being organised by the Department of Scientific and Industrial Research, with the assistance of a local advisory committee under the chairmanship of SIR MAYNARD JENOUR.

We expect some 300 or so delegates from industry and industrial research organisations to attend, and a feature of the programme is the ample time that will be allowed for discussion following each speaker's paper.

The speakers, who will deal with various methods of handling and disseminating information, include DR. R. M. LODGE (Research Director, British Nylon Spinners Ltd.); DR. M. R. HOPKINS (Senior Lecturer, Physics Department, University College, Swansea); MR. W. S. ROBERTSON (Chief Executive Officer, Scottish Council Development and Industry); DR. D. J. URQUHART (Head of the D.S.I.R. National Lending Library for Science and Technology); MR. PERCY CUDLIPP (Editor of *The New Scientist*); MR. EDGAR ANSTEY (Chief Films Officer, British Transport Commission); and MR. L. W. STEVENS-WILSON (Head of Steel Users' Section, B.I.S.R.A.).



# DESIGN AND FABRICATION FOR HEAVY INDUSTRY

Well over half a century of experience in the design and construction of mining plant and buildings, coupled with the ability to design and fabricate specialist equipment—such as rotary dryers, grinding, classifying and pelletising equipment—enables Head Wrightson Stockton to offer an extensive service to the mining, chemical and iron and steel industries throughout the world.

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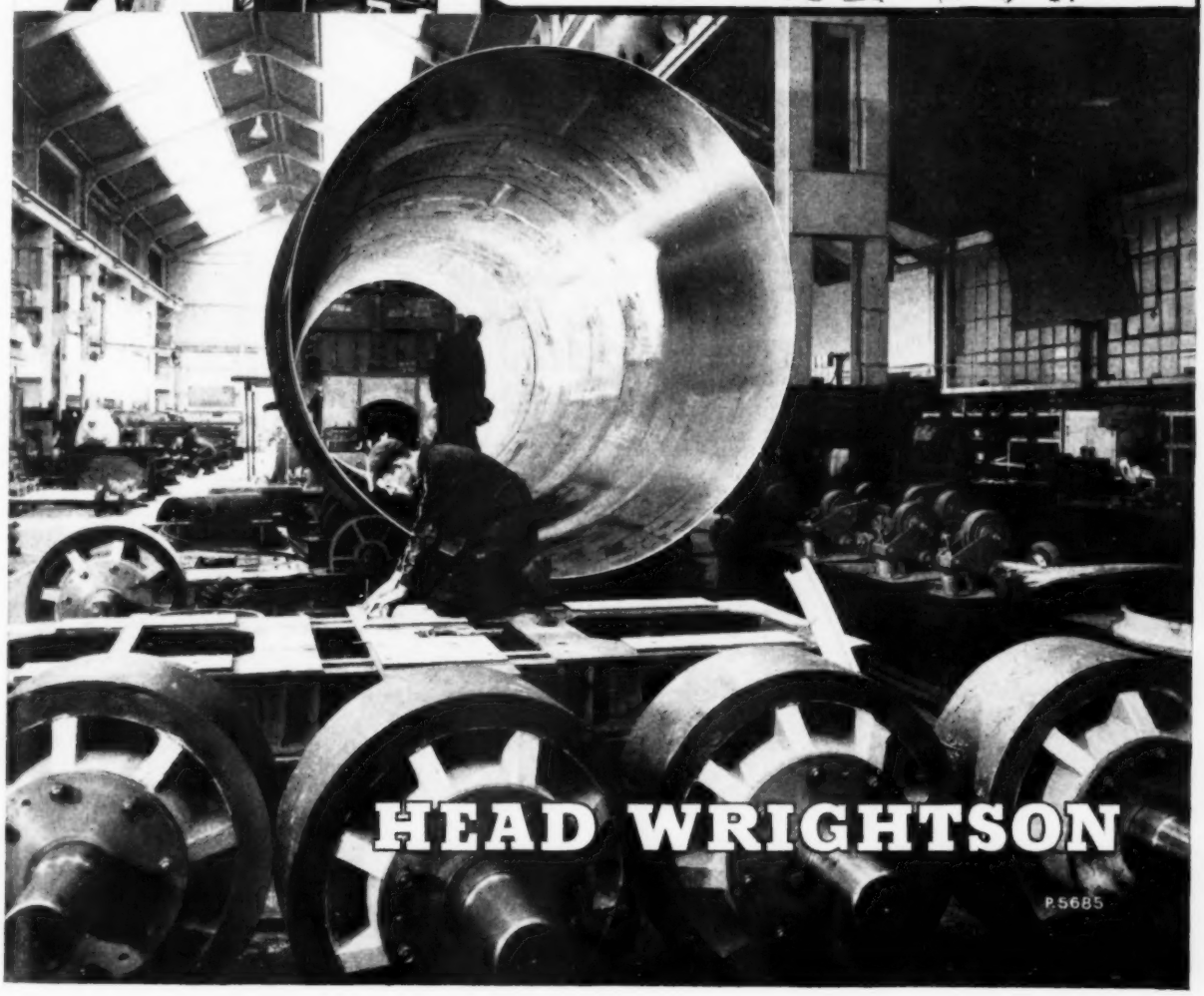
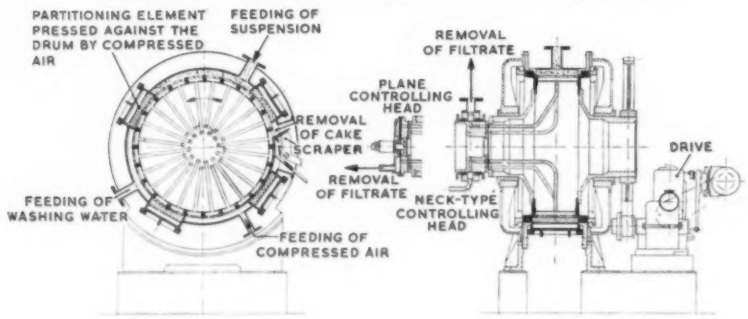


# PLANT EQUIPMENT NEWS

### Rotary Filter for Combined Pressure-vacuum Operation




The ability to operate continuously either at pressure or vacuum, or with both simultaneously, is an unusual feature of the most recent design of "Fest" rotary enclosed filter. In addition to the advantage of operating under a dual pressure régime, this design eliminates the need for a trough and agitator, provides countercurrent washing conditions and fractionation of filtrate, and is specially suitable for separating hot or very cold suspensions with or without volatile components.

This combination of advantages is made possible by the use of partitioning elements, aided by dividing ledges, which, fitted around the periphery of the filter drum, provide the various parts of the filtering cycle. The position of the ledges fixes the cake thickness and the partitioning elements, which slide tightly between the partitions, are pressure loaded, the pressure being regulated either mechanically or pneumatically, according to the working pressure. How the various parts of the cycle are obtained is shown in the illustration. Thus, compressed air and water under pressure can be used for the drying and washing parts of the cycle, in addition to the feeding of suspension under pressure.

The drain pipes from the various compartments may be arranged to end in a controlling head device which allows the main filtrate and the wash filtrates to be collected separately. If several filtrates are required, it is possible to have individual collecting troughs fitted within the filter drum. In this case, however, the filter is used under super atmospheric pressure only.

A further feature contributing to the controllability of the unit is the infinitely variable drive for the drum. The filters can be supplied in the common acid-resistant alloy steels or lined with hard rubber, lead or PVC. **Bayerische Berg-, Hutten- und Salzwerke A.G., Hüttenwerk Sonthofen, Maschinenfabrik und Eisengiesserei.**

**BCE 9109** for further information

### Proportioning Pump and Automatic Sampler for Use with AutoAnalyzer

Development of a two-speed proportioning pump and an automatic sampler of greater capacity that extend the scope of laboratory applications for the AutoAnalyzer is announced by Technicon Instruments.

The Technicon AutoAnalyzer, available in laboratory and plant models, is a continuous flow system that automates each step of a chemical analysis now performed manually, including measuring, mixing, purifying, processing, comparing and recording. Because sample size, time and temperature remain constant, reproducible results can be obtained down to parts per thousand million with a precision of ±1%. Since the AutoAnalyzer checks the sample to be analysed against a continuous stream of standard sample, the process is self-correcting.

Until now, automation of laboratory procedures was practical only for large numbers of repetitive tests, as each test series required long set-up time. With the advent of the AutoAnalyzer's two-speed proportioning pump, set-up time between test series is reduced to as little as one minute.

The new sampler holds 200 test-tubes of 30-ml. vol. on an automatic, self-revolving rack, making it possible to test-run the AutoAnalyzer for more than 30 hr with one rack-loading. The machine can be set at the end of a work day and will continue to operate overnight, unattended, providing 200 analytic results the following morning. The sampler is equipped with two timers—one controlling a selective aspiration period, the other the total cycle-time. The system is cleansed during the difference between the two timer settings. **Technicon Instruments Co. Ltd., 26 Warwick Road, Earls Court, London, S.W.5.**

**BCE 9110** for further information

### A Selective Overhead Conveyor System

Webb Conveyors & Automation Ltd. announce the development of an electronically-controlled overhead conveyor which opens up entirely new possibilities in the realm of mechanical handling.

The system permits the use of a standard overhead conveyor without any of the special mechanical attachments which are normally associated with other types of selective handling equipment. It can be adapted to deal with virtually any number of pick-up points and delivery stations.

A typical system now in course of manufacture involves 30 machines, each of which must be fed with any one of 70 different raw materials. On long continuous runs, some machines use the same material for several weeks, whilst others change their material feed every few hours. The flexibility of this particular system is such that feed variations of this nature can be carried out in less than two minutes by a single operator at a central control console.

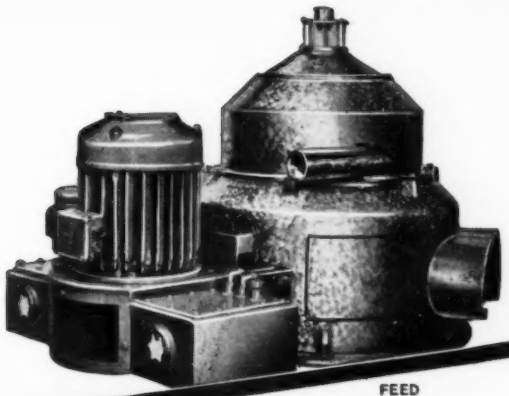
An important point to note is that the operator can change the material feed of any machine during normal shift working without affecting any of the other machines. The procedure simply involves changing a plug from one socket to another, whilst the control centre goes on monitoring the supply of material to all 30 machines. **Elliott-Automation Ltd., 34 Portland Place, London, W.1.**

**BCE 9111** for further information

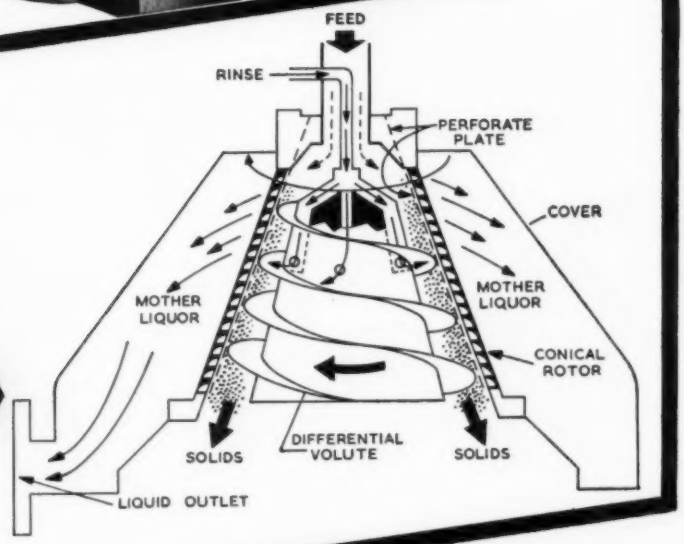
### Mill has Screening Area of 360°

The Stokes tornado mill, of which two models are now available, incorporates a screening area of 360°, which gives it a performance unsurpassed by many other mills.

Particle size is controlled by three factors: size screen, number of blades and speed. Screens range in size from 1-in. perforated plate to 100 wire mesh. The number of blades can be varied from two to fourteen; speed is controlled by a



Now the **Super-Screen** joins the Sharples range

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# PLANT EQUIPMENT NEWS

variable speed pulley, adjustable for 2200, 3600 and 600 rpm.

The mill can reduce materials that are wet, sticky or heat sensitive—always a difficult matter for most types of mill. Briquetted materials, ordinarily difficult to granulate without producing considerable fines and heat-sensitive materials containing oil or fat, are all being successfully processed, in the latter case because of its 360° screen.

In preparing wet materials for drying, its ability to control particle size in the wet stage reduces drying time by as much as 50%, while among the dispersions handled by this machine mill are pigments, food colours, insoluble dyes containing starch, sugar, cement and similar materials —liquid dispersions, such as emulsions and suspensions—and many emulsions that are stabilised with gum or resin. Some of the details of the two machines (models 44-0 and 44-1) available are: horsepowers, 5, 5-25; Rotor diameters (inches), 10, 12; screen areas (sq. in.) 160, 270; and numbers of knives, 14, 16 respectively. **Alchem Processes Ltd., 36-38 Peckham Road, London, S.E.5.**

**BCE 9112** for further information

### Electro-magnetic Valves

A wide range of solenoid-operated or electro-magnetic valves is now available from Londex Ltd.

These valves have uses in almost every industry for such widely differing applications as controlling welding gases and cooling water to controlling the automatic removal of a die by compressed air from pressed components.

The valves are normally fitted with brass bodies, but stainless steel or plastic models can also be supplied and all A.C. and D.C. voltages can be catered for.

Models for continuous high-speed operation, up to 48,000 movements per hour, can be supplied, and valves which will close against a pressure of up to 1400 psi are also available.

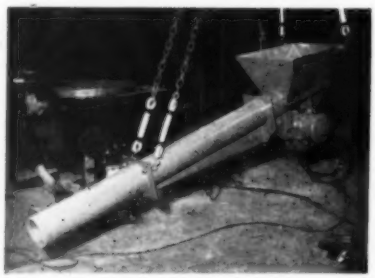
There are five basic models of valve in the range, four of which are of the single-way electro-magnetic variety; the fifth is a three-way and four-way valve for use with compressed air and hydraulic fluids. The four models of single-way valves are designed respectively for use with air, water and non-corrosive gases and liquids up to a viscosity of 6° Engle, for low gas pressures, for steam, and for rapid and/or frequent operation.

For those applications where the loss of head across a valve must be kept to a minimum, models are available which have the diameter of the internal port diameter equal to that of the pipe.

The range of valves is manufactured by Concordia G.m.b.H., Stuttgart. **Londex Ltd., Anerley Works, 207 Anerley Road, London, S.E.20.**

**BCE 9113** for further information

### Suspended Type Tubular Feeder



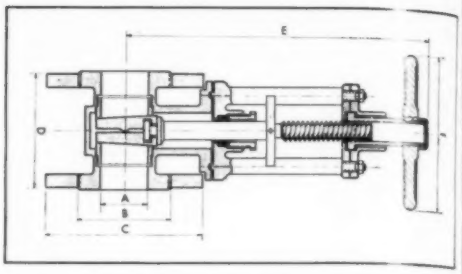
This newly developed, suspended type, vibrating feeder is completely self-contained, being operated from a rotary electric vibrator. The conveyor illustrated is 6 ft long and has a 6 in. dia. feed tube. When operating at an angle of approximately 10° it will feed 8-10 tons per hr and of dry sand having a bulk density of about 100 lb/cu ft. Within the range of tubular feeders, units are being produced with tubes ranging from 3 in. dia. up to 10 in. dia. and from lengths of 3 ft to 10 ft. **Blending Machine Co. Ltd., Bond Street, Hockley, Birmingham, 19.**

**BCE 9114** for further information

### Stainless-steel Gate Valves

A new range of flanged or screwed stainless-steel gate valves is now being produced by the French concern, Gachot. An 18/8/3 Mo stainless steel is used in their construction and it is used economically, in the case of the flanged models, by employing threaded portions at the ends, on which are screwed mild-steel bolting flanges. The stainless-steel joint faces stand proud of the m.s. flange faces.

The type 501 range has double wedges, renewable seats, and PTFE packing in the stuffing box. In addition, an opening indicator is provided. Valves in this




range, available in sizes 1-2½ in, can be supplied with either pneumatic or electric controls.

A similar model is available in 3-10-in. sizes, removable seats being provided in the models up to 6 in. and those above this size having machined seats. **Gachot A.G.-B.P. 16, Enghien (S.-&-O.), France.**

**BCE 9115** for further information

### Multi-stage Liquid-liquid Extractor

Intended primarily for pilot-plant studies and for small-scale production, a multi-stage mixer settler unit now in production approaches closely the requirements of an ideal pilot extractor; it permits the experimental determination of both the equilibrium and operating lines for any type of liquid-liquid extraction process.

It consists of twenty shallow mixer-settler compartments placed one upon another, each stage being provided with a reciprocating perforated plate agitator and with heat transfer plates interposed between pairs of stages.

The heavy phase is fed at the top of the assembly and the lighter phase at the base, the flow being caused by the gravity differential between phases. An important feature is that uniform mechanical mixing prevents phase by-passing, and mixing intensity can be varied to give mixing without emulsification. Although the flow between stages is counter-current, the flow through the mixing zones is co-current, with the result that efficiencies approaching very closely to 100% are obtained without the possibility of the stage efficiency exceeding 100%.

Other important features include a low hold-up to throughput ratio, and short times only to attain equilibrium are required.

The heat transfer plates also provide close control of temperature as well as any desired temperature pattern. Finally, the unit is so arranged that feed or solvent can be injected into any stage, allowing rapid change in the number of operating stages and the distribution of enriching and stripping stages. **The Pennsylvania Tool & Manufacturing Co., Hartman and Wellington Streets, York, Pennsylvania, U.S.A.**

**BCE 9116** for further information

### New Range of Welding Positioners

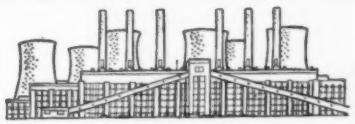
These machines are made in Germany for the British market and ten models cater for maximum loads of 33 lb up to 10 tons. The bench model DB15 (illustrated in top picture) is the smallest of the range, having a capacity of 33 lb. It has an infinitely variable speed range of

BCE 9066 for further information

# THE ACTIVITIES OF THE PRODORITE ORGANISATION are concentrated on all problems concerning chemical corrosion and mechanical wear and tear:—

## 1 CONTRACT WORK

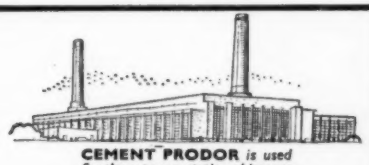
concerned with chemical engineering; acid proof constructions and industrial finishes; floor laying; complete plant design, installation and maintenance; effluent disposal and drainage schemes, carried out by our own personnel using Prodorite products.



*Extensive sub-contracts in C.E.A. Power Stations*

## 2 CHEMICAL RESISTING CEMENTS

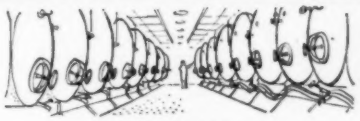
and compounds for all types of industrial application. These cements fall into two basic categories: silicate based and resin based; the two combined ensuring the availability of a suitable cement to fill every anti-corrosive need.



**CEMENT PRODOR** *is used for these power station chimneys*

## 3 PROTECTIVE COATINGS and LININGS

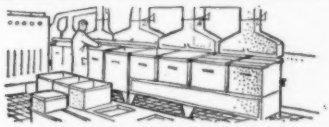
to storage and transport tanks and pipelines; for industrial processes; and linings to vessels for foodstuffs, beverages and aviation fuels. Available for bonding or spraying on site in a variety of forms.



**PRODOR-GLAS** *lined cold room tanks*

## 4 PLASTICS FABRICATIONS and RESIN/GLASS LAMINATES

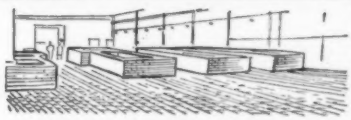
Rigid P.V.C.; plasticised P.V.C.; polythene in welded sheet form for fume ducting, tank linings, fans etc; for chemical, plating, pickling and allied industries. Complete range of ORGLAS laminates having highest possible chemical and temperature resistance. Wide industrial applications, including the food industry.



**P.V.C.** *fabrication scheme for fume extraction*

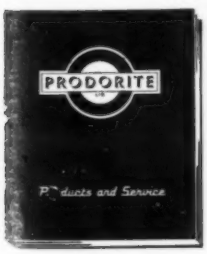
## 5 INDUSTRIAL WALL and FLOOR FINISHES

including acid resisting and heavy duty types in the form of paviors, tiles, flags, and insitu, with suitable additives. Also decorative wall and floor treatments for use in flats, hotels, public buildings, etc.



*Corrosion resisting floors and walls in Acid Pickling shop*

The products and services of the Prodorite Organisation extend from the manufacture and supply of materials to complete constructions carried out by the Contracting Department to customers' requirements. Prodorite products give complete protection from atmospheric conditions, chemical corrosion and mechanical wear and tear of any sort.




**SEND FOR YOUR FREE COPY OF THE PRODORITE BOOKLET**

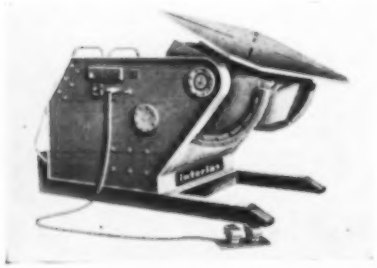
This stiff-cover publication contains 25 pages of invaluable information about the products and services of the Prodorite Organisation. It includes photographs of actual installation and details of the Prodorite research, development and consultation services. Technical information about individual products is also available in leaflet form. Please quote publication reference number: P 103

Write to: **PRODORITE LIMITED** (Chemical Engineers and Consulting Contractors) **EAGLE WORKS, WEDNESBURY, STAFFS.,** or Telephone Wednesbury 1821 (10 lines).

# PLANT EQUIPMENT NEWS

0.3-3 rpm with a foot-operated remote control so that the operator has both hands free for welding.

At the other end of the range the 10 ton model DB 10000 (illustrated in lower picture) has a speed range of 0.06-1.2 rpm. Between these models come positioners for maximum loads of 2 cwt, 5 cwt, 1 ton, 1½ tons, 2½ tons, 3½ tons and 6 tons.



Complete jigs for the automatic welding of small cylinders are also obtainable consisting of rotating equipment and supports for the welding of lead with all necessary electronic controls. **Interlas Ltd., 9 Church Street, Ampthill, Bedford.**

**BCE 9117** for further information

## The DCL "S" Micro Pump

A metering pump which has achieved remarkable popularity since it was first introduced is now available in a micro model in a range of capacities—the smallest from zero to 100 cc/hr, the largest from zero to 1500 cc/hr—controlled by micrometer adjustment. With this model the standard plunger heads are constructed in 18/8/3 stainless steel, and diaphragm heads in a wide range of materials are also available.

Drive is by two-speed synchronous motor which for safety operates from a 25/30 V supply, and where such a supply is not available, a small portable plug-in type transformer is supplied to enable the equipment to be used from any normal a.c. voltage.

The simplex pump can be used with one head and can be converted into a duplex pump by adding another head. If desired, the heads may be used independently: price is £75 (simplex) and £99 5s. (duplex) and £4 10s. for the transformer. **The Distillers Co. Ltd., Great Burgh, Epsom, Surrey.**

**BCE 9118** for further information

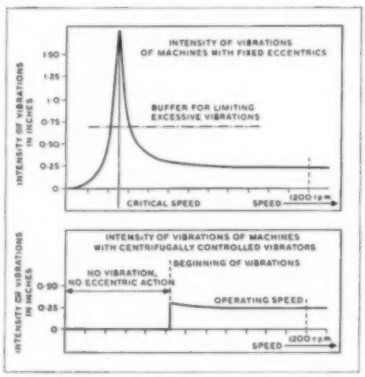
## Combined Vibrating and Lifting Machine

Large moulds for pre-cast concrete units or bulk handling containers can readily be vibrated with a new combined machine known as the "Vibra Lifta". If a container is mounted on supports giving a minimum ground clearance of 16 in., the machine can be wheeled under the container. The latter is raised by the steering handle and is then ready for filling and vibrating. After vibration has been applied, a hydraulic cylinder acting as a release check enables the load to be lowered safely and gently back on to its supports. The vibrating motor is safely housed under the table away from spillage or accidental damage. In one application a 25-ft-long steel rafter mould was successfully vibrated by two 1-ton capacity units. The weight of the mould was 15 cwt empty and nearly 2 tons filled. All standard units are fitted with 2 ft × 2 ft vibrating platforms. Their height lowered is 15 in. and height elevated 17 in. **Triton Engineering Co. (Sales) Ltd., Sydenham Road, Croydon.**

**BCE 9119** for further information

## Centrifugally Controlled Vibratory Screen

In a notable continental design of screening machine its vibratory motion is readily adjustable to the properties of the material to be screened. The body of the machine is subjected to circular vibrations, the movement being controlled so that, when starting or stopping, the critical speed is passed with little or no eccentric motion. At full speed the vibrators and the body of the screen form a freely vibrating body in which the effects of the moving mass are compensated by the vibrators; the vibrations are therefore not transmitted to the building in which such a machine is installed, hence special supporting structures are unnecessary.

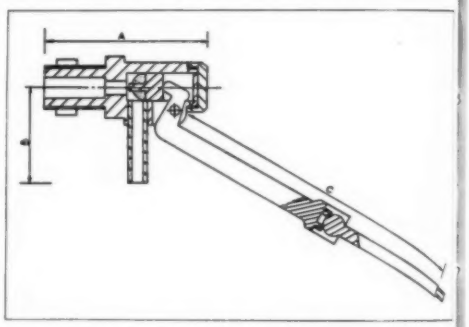



The action of these vibrators, produced by Gutehoffnungshütte, is illustrated below and compared with rigid vibrator machines. Fig. 1 shows the vibrating intensity of a rigid type of machine plotted against driven speed. In starting and stopping the intensity of the vibration causes the transmission of shocks to foundations, etc. Fig. 2, on the other hand, shows the same plot for the special design. The resonance range is passed without eccentric action and only when the critical speed is passed does the eccentric come into play and cause the screen to vibrate in uniform circles. Variations of speed have no effect on the intensity of vibration, and neither is there any inadmissible eccentric action when the motion of the machine ceases. This is because the eccentrics are automatically brought to rest before the critical speed is reached.

The screen is available in single-, double-, three- and four-deck models, the projected areas per screens varying from $31\frac{1}{2} \times 63$ in to $49\frac{13}{16} - 197$ in. **Gutehoffnungshütte Sterkrade Aktiengesellschaft, Dusseldorf Works, Dusseldorf-Grafenberg.**

**BCE 9120** for further information

## "Polythene" Adjustable Ball Valve




Illustrated is the "Barflo" high-density all-plastic ball valve with an all-direction adjustable arm. Designed to withstand the test requirements of B.S. 1212: 1953 (for metal valves), it will not corrode and is unbreakable. There are no metal parts. ½-in. and ¾-in. BSP sizes available. **Barflo Ltd., 56 Cavendish Place, Eastbourne, Sussex.**

**BCE 9121** for further information

## Flow Indicators for Opaque Liquids

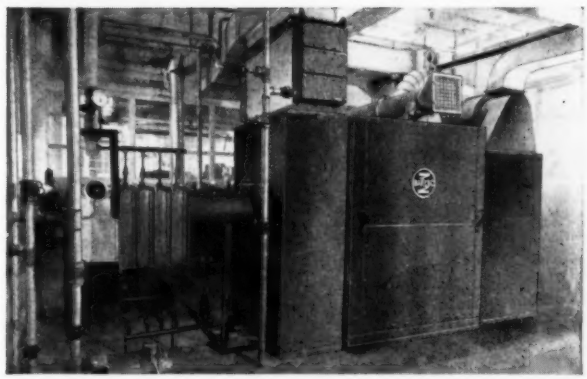
The problem of observing the flow of an opaque liquid through a pipeline has been minimised by the introduction of a range of Magnetel flow indicators.

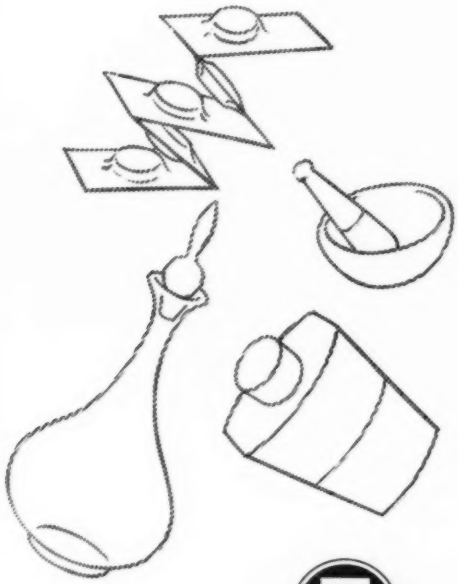
These instruments, which can be used with liquids of almost any viscosity—such as thick oils—are equally suitable for indicating flow either horizontally or vertically upwards.

The internal parts are readily accessible for cleaning while the body is still in the line, and can be turned through 90° if

# DRY HYGROSCOPICS

*Model CO Dehumidifier installation at Reckitt & Sons Limited, Hull, for their Steradent process room*





Many chemicals and pharmaceutical products are harmed by atmospheric moisture. Damp weather may spoil both the product and the smooth flow of the production processes if atmospheric humidity throughout the plant is not controlled. Birlec Direct Dehumidifiers offer a simple and highly efficient means of holding the relative humidity of a process room or storage enclosure at a safe low level.

SM/B6231

**AEI-Birlec Limited**

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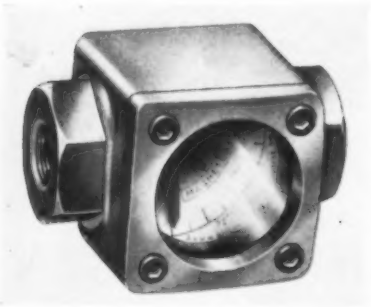
# PLANT EQUIPMENT NEWS



required to change the direction of flow. It is easily adapted for extremely low flow rates, and the smallest change in flow is shown against the scale. It will also accept a wide range of capacities when full flow is shown on the scale.

A switch Magnetel is also available which operates a switch automatically to give an alarm when the flow is interrupted.

The Magnetel is available in standard ½-in., ¾-in., 1-in. and 1½-in. sizes, with ends screwed B.S.P.T., with adaptors to match ⅜ in. and 1¼ in. The 2-in. size has flanged ends.

Standard sizes are available ex-stock; other models on short delivery. **Sir W. H. Bailey & Co. Ltd., Albion Works, Patricroft, Manchester.**

**BCE 9122** for further information

### New Ceramic Insulating Brick Withstands Temperatures to 4200°F

The Ipsen Insulating Ceramic Brick (I.C.B. 4200) recently developed will withstand temperatures of 4200°F. It is formed in standard brick size of 2½ in. × 4½ in. × 9 in. It has a mean thermal conductivity of less than 1.0 at 2200°F. This is three times the insulating value of a 2600 I.F.B. Each brick weighs about 2 lb and has an 89 per cent porosity which will permit the passage of liquid. In nitrogen, hydrogen or oxidising atmosphere its temperature limit (hot face) is 4200°F. In a reducing atmosphere with carbon present or in contact with $SiO_2$ or $Al_2O_3$ it is 3000°F and in contact with SiC it is 2200°F. It will have important applications in the nuclear and metallurgical fields. **Ipsen Industries Inc., P.O. Box 500, Rockford, Illinois, U.S.A.**

**BCE 9123** for further information

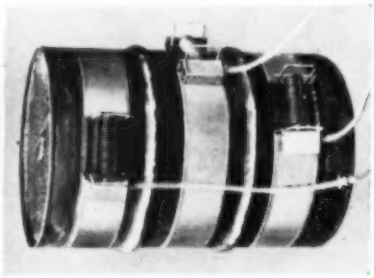
### Visible and Ultra-violet Spectrophotometer

This new spectrophotometer is designed as a high-performance, low-cost, double-beam recording instrument for use in the ultra-violet and visible regions of the spectrum. It is similar to the existing Perkin-Elmer Infracord spectrophotometer for use in infra-red regions. Importance is attached to attaining good photometric accuracy and reproducibility, together with low values of stray light. It incorporates automatic gain control and source switching. High resolution is achieved at low noise levels, e.g. 0.2 mμ at 250 mμ with a noise level of less than ½ per cent. Scanning time is 2 min in each region, or 8 min for particularly sharp absorption bands. **Perkin-Elmer Ltd., Beaconsfield, Bucks.**

**BCE 9124** for further information

### Electrothermal Drum Heater

This new device is a quick, economical and portable means of heating drums containing viscous or solidified materials in factories. By using single or multiple



heaters it is possible to apply heat to any or all sections of the drum. A drum with three heaters is illustrated. The heaters may be used on the drums standing upright, lying on their sides or suspended. The handles, which remain cool during operation, are joined by a strong steel spring which ensures intimate contact with the drum. **Electrothermal Engineering Ltd., 270 Neville Road, London, E.7.**

**BCE 9125** for further information

### Iris-diaphragm Control Valve

A new type of regulating valve makes use of the iris-diaphragm principle and overcomes one of the main disadvantages of other types of valve by ensuring that the orifice is always symmetrical to the flow axis. The advantages of this type of valve are that it prevents clogging when solids are present and cavitation effects are minimised.

The diaphragm segments are designed to provide a valve which is self-cleaning in either direction of flow. Indicators provided with these valves give a precise reading of the percentage of the cross-sectional opening. Materials of construction include all the common metallic materials including the austenitic steels and nickel alloys. Sizes, of which there are seven, range from 25 to 250 mm and they are suitable for the operating conditions of pressure and temperature commonly encountered in industry. **Emile Egger & Co., Cressier, Switzerland (U.K. agents: Moffat & Bell Ltd., London).**

**BCE 9126** for further information

### High-speed Impulse Switch

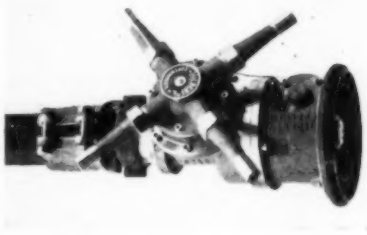
This new electro-mechanical switching unit is designed to provide electrical circuit switching from the rotation of a shaft by means of cam-operated heavy-duty switches.

Intended for use with the series F43 high-speed electromagnetic counter unit, an alternative form of the switch with a different cam-form is particularly suited to limit switching on rotary valve actuators and similar applications. **Lancashire Dynamo Electronic Products, Rugeley, Staffs.**

**BCE 9127** for further information

### Industrial Tank Cleaner

The principal component of a tank-cleaning tool is a water-driven turbine which rotates the cleaning nozzles through reduction gears. The cleaner can operate from any position and provides its most effective and economical cleaning action at a pressure of approximately 115 psi. This pressure will provide a jet of



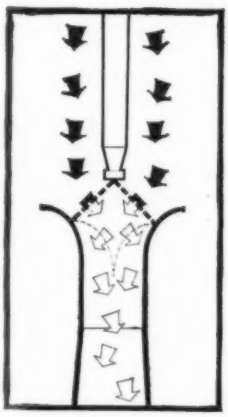
water 80 ft high, but it is possible to use the cleaner with other fluids. **C.P. Equipment Ltd., Mill Green Road, Mitcham, Surrey.**

**BCE 9128** for further information

### Spray-drying Plant

The Anhydro spray driers now being manufactured in Britain by A.P.V. Ltd. employ a stainless-steel centrifugal atomiser. In these plants air for drying can be supplied by indirect steam heating or by indirect oil-fired heaters, and, in addition, the units can be supplied with-

# 'DOUBLE SCRUBBING' IS THE ANSWER



The first cone of water removes the coarser solid particles; the second cone ensures that even the very finest are collected. The position of the water injection nozzle can be varied to obtain a droplet size to suit *any* set of conditions.

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In the Ambuco venturi scrubber, made under licence from Waagner-Biro, the gases are compelled to pass through *two* complete cones of water. This exclusive feature guarantees up to 99·6% collection efficiency of most entrained solids in the range 5 to 0·01 microns. **Send for fully descriptive bulletin.**

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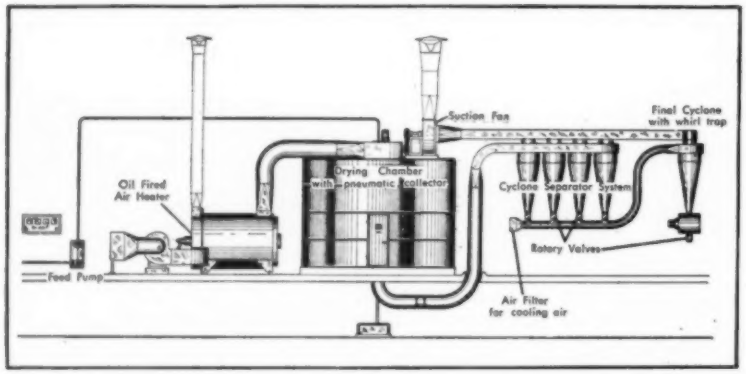
# PLANT EQUIPMENT NEWS

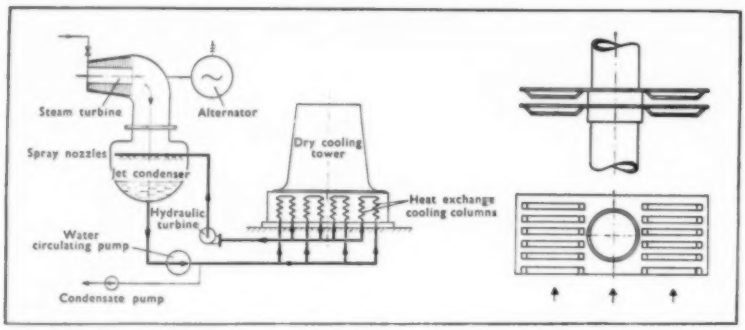



out heating for use as spray crystallisers. In the case of the oil-fired units automatic control of the air temperature is provided for temperatures up to 250°C, whereas where lower temperatures are acceptable steam heating, similarly controllable, is employed. Collection of product is by groups of cyclones of high separating efficiency for particles of 8 microns and above. The dried product is cooled by a special air cooling arrangement fitted downstream of the cyclone rotary valves. This ensures that the discharged material does not exceed the temperature of the cooling air by more than 5°C.

Capacities, based on steam at 85 psi, an outlet temperature of 80°C for the drying air and an inlet temperature of 156°C, are from 45-2240 lb/hr. In the case of oil-fired heaters, for an inlet air temperature of 215°C and an outlet temperature of 90°C, the capacity range is 180-4500 lb/hr. **A.P.V. Ltd., Crawley, Sussex.**

**BCE 9129** for further information

## Dry Cooling Tower System




A recently evolved system for condensing the exhaust from steam turbines has, as one of its main elements, a jet condenser. The water supply to this unit circulates through an air cooler, with the excess water arising from the condensation being passed back to the steam plant. A pump circulates the water through the heat exchanger system, and after passing through the latter, the water expands through a turbine and thence through the spray condenser nozzles. The power generated in the water turbine contributes to the power required for circulating the cooling water.

A special heat-transfer surface is employed in the heat exchangers. The material is 99.5 per cent purity aluminium and the extended surface is slotted to form small strips. An important feature of this design is that it prevents the development of a thick boundary layer at the surface and is therefore conducive to good heat transfer at low flow rates Another property of this surface is its comparatively low pressure loss. The heat transfer surface is provided in packaged groupings which are readily fitted into either natural-draught or induced-draught towers.

The principal advantages of the combined system are: the absence of fog or ice formation through the elimination of the wet cooling tower; absence of feed water contamination through the condenser; and, in the case of nuclear power stations, the completely closed cooling system will prevent contamination of the surroundings by radiation.

Normally these complete condensing units are available in 5-150 MW capacities. This equipment, known as "The System Heller", is supplied by **Komplex, Budapest, Hungary.**

**BCE 9130** for further information

## New Publications

*Photo-electric Control Equipment*

Data Sheet PR, Issue 3, describes three types of photo-relay, together with associated light sources and viewing heads. In each equipment the relay is actuated by a change in the intensity of the light falling on a photocell; applications include counting; position inspection; sorting by size or mark; conveyor monitoring for over- or under-loading; off- or on-loading control; and elevator or door-opening control. Relay PR6 responds to light level; PR7 to a pulse of light; and PRD2 combines a photo-relay (responding to light level) and a delay switch for relatively slow-speed conveyor control. The latter may be used to give relay operation when a sustained accumulation or sustained cessation of objects occurs on a line.—**Elcontrol Ltd., Wilbury Way, Hitchin, Herts.**

**BCE 9131** for further information

*Gas Respirators*

Three leaflets, RES/1-3, describe "Puretha" and "Gaspro" respirators. The former has a separate canister, connected to the facepiece by a rubber tube, and harness is available so that the canister may be worn on the chest, back or carried at the side in a haversack. Sixteen types of colour-coded canisters are produced for protection against low concentrations of particular types of gas or vapour. The "Puretha" Vista full-face mask gives a wide range of vision and a completely gas-tight fit; alternatively, mouthpiece, nose-clip and goggles can be supplied. The "Gaspro" respirator has a smaller canister (with a third of the lifetime) attached to a moulded-rubber facepiece. —**Siebe, Gorman & Co. Ltd., Neptune Works, Davis Road, Chessington, Surrey.**

**BCE 9132** for further information

*Electrical Contact Controllers*

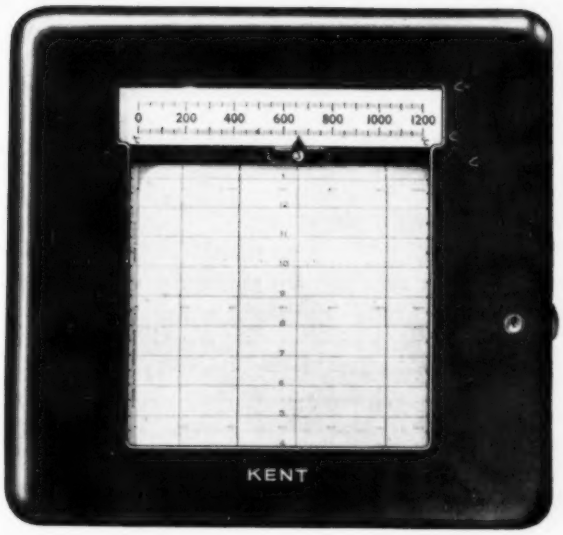
These instruments operate a relay according to the conditions (e.g., temperature or pressure) in the plant controlled; the relays can actuate valves, pumps, alarm systems, etc. Both indicating and chart-recording types are described in a 4-page leaflet, No. R/43. The recorder can be supplied with a duplex system, incorporating two relays, to control two separate conditions in a plant or conditions in two separate plants.—**Negretti & Zambra Ltd., 122 Regent Street, London, W.1.**

**BCE 9133** for further information

*Light-weight Oxygen Breathing Apparatus*

Details of the "Stelox" half-hour oxygen breathing apparatus are given in a leaflet. The cylinder, reducing valve, "Protosorb" canister, breathing bag and relief valve are contained in a fibreglass case worn on the chest. The total weight, with cooler, is 12 lb.—**Siebe, Gorman & Co. Ltd., Neptune Works, Davis Road, Chessington, Surrey.**

**BCE 9134** for further information




## Now even better—

- Available now for all millivoltage and temperature measurements, in single- and multi-point form; also for oxygen-percentage and conductivity measurement
- 1-second or 2-second full-scale pointer travel—deadbeat, no "overshoot"
- Entirely mains operated
- Chart speed variable from ¼in./h to 4in./min
- High-gain amplifier covering wide range of a.c. and d.c. inputs
- Sealed servo-motor unit with built-in tachogenerator
- Plug-in units and interchangeable sub-assemblies

# The New mark 3 multelec electronic strip-chart recorder

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**The *Type 3* Reference Unit**

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# PLANT EQUIPMENT NEWS

*Second Mask for Compressed-air Breathing Apparatus*

The second mask, described in leaflet BA/2, enables two persons to breathe air from the one cylinder of a self-contained breathing apparatus. The attachment is especially valuable in rescue operations, and does not prevent use of the set by a single person. It can be fitted to existing breathing apparatus. **Siebe, Gorman & Co. Ltd., Neptune Works, Davis Road, Chessington, Surrey.**

**BCE 9135** for further information

*Electrical-wire Enamels and Insulating Varnishes*

An 8-page booklet, "Wire Enamels and Insulating Varnishes for the Electrical Industry", contains technical details and a pull-out selector chart in which insulating varnishes and winding wire enamels are identified by thermal classification. — **Schenectady-Midland Ltd., Oldbury, Birmingham.**

**BCE 9136** for further information

*"Impalco" Aluminium Technical Data*

Detailed information is given in a 69-page booklet on three grades of commercially pure aluminium, seven non-heat-treatable aluminium alloys, nine heat-treatable alloys and five other alloys for special purposes. Weight tables are included for plate, sheet, strip, bar and tube material; other tables deal with composition limits, mechanical properties and a comparison with other metals.—**Imperial Aluminium Co. Ltd., P.O. Box 216, Witton, Birmingham, 6 (or I.C.I. sales offices).**

**BCE 9137** for further information

*Resins for Rubber and Adhesive Compounding*

Phenolic and terpene-phenol resins are dealt with in a 12-page booklet, "Resins for Rubber Compounding and Rubber-based Adhesives". In the first part of the booklet, on rubber compounding, sections are devoted to functions and types of phenolics, compounding with phenolics and applications; the adhesives portion of the booklet covers types of resin, modification of base elastomers, adhesive preparation methods and applications. Properties of the resins are tabulated. — **Schenectady-Midland Ltd., Oldbury, Birmingham.**

**BCE 9138** for further information

*Aluminium-alloy Profiled Building Sheets*

A 19-page booklet, " 'Impalco' Profiled Sheets for Building", gives descriptive information, technical data on corrugated and troughed building sheets, and illustrations of industrial uses. General notes are included on installation, painting (this is not normally necessary) and storage.—**Imperial Aluminium Co. Ltd., P.O. Box 216, Witton, Birmingham, 6 (or I.C.I. sales offices).**

**BCE 9139** for further information

*Current Overload and Underload Relays*

Data sheet OR/UR, Issue 2, describes relays suitable for use in mechanical handling or processing equipment to stop an electrically-driven unit when it becomes overloaded, and restart it when normal conditions are restored. In each case the relay is connected to the secondary of a transformer, the primary being in circuit with the controlled load. If, for example, a crusher is being fed with material from a conveyor, over-feed will result in the crusher motor current rising; when the current reaches a preset value, the overload relay operates to stop the feed conveyor. As the crusher clears itself, the motor current falls, and again at a preset value the underload relay can operate to restart the conveyor motor. The trip point of both forms of relay can be set at any point between 20 and 120% of full load current. A signal lamp indicates when the monitored current is within the desired limits.—**Elcontrol Ltd., Wilbury Way, Hitchin, Herts.**

**BCE 9140** for further information

*Automatic Weight Control*

Data sheet AW, Issue 4, describes trip point controllers for use with load cells in the electronic weighing of liquids and bulk materials such as chemicals, ores, foodstuffs, cement, clay and finished products. The load, such as a tank, hopper, conveyor section, truck or loading platform, is supported on one or more load cells, which use strain gauges to give an electrical indication of change of load. The trip point controller, type LC4, when connected to the load cells, can be accurately set to operate a relay at any point from zero to full load on the load cells. The relay in turn can control the operation of pumps, conveyor motors, hopper gates, etc., to give weight control. In the LC5 unit, facilities are arranged for the operation of one or more remote deviation indicators which show the percentage approach of the trip-point condition, i.e., 100% deviation at zero load and 0% deviation at the trip point. Load cells are produced to meet specific requirements, covering loads ranging from 20 lb to 2000 tons.—**Elcontrol Ltd., Wilbury Way, Hitchin, Herts.**

**BCE 9141** for further information

*Dust Respirators*

Two dust respirators described in leaflet DUS/1 fit over the nose and mouth, leaving vision unrestricted. The V.H.E. respirator is suitable for use in conditions where very fine dusts are present, and is specially recommended for lead-burning operations. The Mark VIII respirator employs a cotton prefilter and a second micro-filter of resin-impregnated merino wool; both filters are quickly replaceable.—**Siebe, Gorman & Co. Ltd., Neptune Works, Davis Road, Chessington, Surrey.**

**BCE 9142** for further information

*Protective Clothing*

A leaflet describing the "Jeltek" range of protective clothing has been issued. Four types of material are used: PVC-coated rayon for the "Standard" range; PVC-coated cotton duck for the "Duck-Dri" range; nylon-based neoprene-coated material for the "Neoprene" range; and PVC-coated nylon for the "Hydex" range. Garments available include long coats, jackets, trousers, leggings and sou'-westers with, in the "Hydex" range, aprons and weathersuits, and in the "Neoprene" range, boilersuits. A further leaflet describes "Jeltek" duffle coats and donkey jackets; the latter are available with leather or PVC panels on the back, shoulders and sleeves. A photostatic branding service is available for the reproduction of lettering, emblems or insignia on any of these garments. A separate price list is issued covering the full range of protective clothing, duffle coats and donkey jackets.—**Jeltek Ltd., Green Lane, Hounslow, Middlesex.**

**BCE 9143** for further information

*Water Treatment Plant*

A 6-page leaflet entitled "Filtration Plant" describes Head Wrightson-Belco gravity, pressure and diatomaceous filters. These are manufactured under licence from the Belco Industrial Equipment Division of Bogue Electric Manufacturing Co., New Jersey, and are available both custom-built and as standard designs. By suitable combinations of equipment, treatments can be provided to suit almost all process water demands. Head Wrightson-Belco deaeration equipment is the subject of a further leaflet. Diagrams and brief details are given of three-stage spray-type heaters, spray-type open heaters, vacuum deaerators and marine deaerators.—**Water Treatment Division, Head Wrightson Processes Ltd., 20-24 Old Street, London, E.C.1.**

**BCE 9144** for further information

*Air-operated Regulating Units*

Air-operated valves, servo-motors and valve positioners are described in a 16-page booklet, No. R30/RU. These valves are for insertion in a pipeline to regulate the flow of gas, hot water, steam, etc., to maintain stable conditions in a plant. The booklet covers diaphragm-operated valves, butterfly, spring-closing and needle valves, diaphragm-lever and servo-motors, proportional cylinders, valve positioners and diaphragm-operated butterfly valves. —**Negretti & Zambra., 122 Regent Street, London, W.1.**

**BCE 9145** for further information

*Instruments for Australia*

A summary of the full range of Negretti & Zambra instruments is given in a 12-page brochure, No. F25A/1. It covers the measurement of temperature, pressure, level, volume, flow and humidity, with separate sections on automatic controllers, meteorological and aeronautical instruments.—**Negretti & Zambra (Australasia) Pty. Ltd., 336 Victoria Parade, East Melbourne, Victoria, Australia.**

**BCE 9146** for further information

# Mr HAYWARD TYLER

TALKS ABOUT

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*A primitive sacrificial rite?*
*An early whistling kettle?*
*An Ethiopian Ernie?*
*Mr. Hayward Tyler wouldn't know*
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*Throughout the 19th century*
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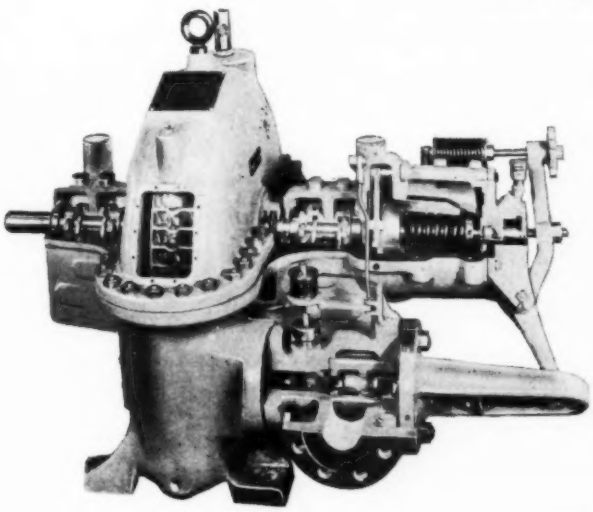

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| Class | Standard | Medium Temp. | High Temp. | |
| Maximum Operating Conditions: | | | | |
| Inlet steam pressure, P.S.I.G. | 250 | 250 | 450 | 450 |
| Exhaust steam pressure, P.S.I.G. | 50 | 50 | 60 | 60 |
| Steam temperature, °F. | 4500 | 4500 | 4500 | 4500 |
| R.P.M. | 5 to 150 | 5 to 150 | 5 to 150 | 75 to 650 |
| H.P. Range | | | | |

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# World of Chemical Engineering

## New Processes

**A new sulphonation process** was presented as a paper by A. DAVIDSOHN, the inventor, at the Third International Congress on Surface Activity, held during September 1960. The principle of the process is to transform an exactly dosed amount of molten sulphur. It is burnt in a stream of silica-gel-dried air and transformed into a dilute $SO_3$-gas stream. This gas is cooled and used for the direct sulphonation of alkyl-benzene, fatty alcohols, etc., in a specially designed sulphonation plant. No oxidation side reactions occur. The products are practically salt-free, odourless and of excellent colour. The only materials entering the process are sulphur and the material to be sulphonated.

**The Allied Chemical Corp.** have acquired Snia Viscosa's new process for the manufacture of Caprolactam. Starting with raw material toluene instead of phenol, the addition of this process to Allied's complex is expected to provide them with broad flexibility in their operations, including the production of derivatives and intermediates other than Caprolactam. Caprolactam is the monomer for nylon 6, and new applications for nylon 6 moulding compound are expected.

**The production of urea** by a new, more efficient, lower cost process has been perfected by the Chemical Construction Corp. of New York. The process employs the principle of carbamate solution recycle, which facilitates the complete consumption of the ammonia and carbon dioxide used as raw materials. Nitrogen fertiliser manufacturers will be afforded continuous, economical and trouble-free operation.

### Britain's First Polypropylene Plant on Stream

I.C.I.'s new polypropylene plant—the first in Britain for bulk manufacture—is now on stream at their Wilton works in Yorkshire. The new manufacturing unit, which has a capacity of 11,000 tons a year, will produce their own brand of polypropylene called "propathene".

The building of the main plant could not begin until I.C.I. obtained a licence under the Montecatini and Montecatini/Ziegler U.K. patents covering the production and use of polypropylene. Work started as soon as this was obtained, and was completed on schedule in sixteen months—a great achievement. The photograph is of the control room.

### First U.K. Production of Sodium Chlorite

Production of sodium chlorite has now started at the Luton plant of Laporte Chemicals Ltd. The Kesting process licensed by Elektro-chemische Werke Munchen is employed. The photograph shows a reaction cascade.

The chlorite will be marketed as a crystalline powder with the normal industrial strength of 80 per cent.

This is the first chlorite plant to go into production in Britain. Previously it has been imported from Europe. The plant is largely constructed of plastic glass, ceramic, stainless steel and titanium metal.

## New Plant

**I.C.I. Heavy Organic Chemicals Division** are to increase their capacity for the manufacture of alcohols by the carbonylation of olefinic feedstocks. New projects will together raise I.C.I.'s total carbonylation alcohols capacity to over 140,000 tons per year.

**The Monsanto Chemical Co.** plans to construct two more plants in New Jersey as part of a plastics raw material and industrial chemical complex. One unit will produce a plasticiser trade-marked Santicizer 160 and the other unit will produce benzyl chloride, a basic ingredient of Santicizer 160.

**The Shell Chemical Co. (U.S.A.)** have acquired a site in Ohio for a new synthetic rubber plant to make polyisoprene. Capacity will be about 36,000 tons a year and production is expected to begin by the end of 1961. Polyisoprene is chemically the same as natural rubber.

**Laporte Industries Ltd.** have reached agreement with the Western Australian Government for the establishment of a £A4-million titanium oxide plant to be constructed at Bunbury, 115 miles from Perth. Production capacity will be 10,000 tons per year and should start by 1964.

**British Geon Ltd.** are planning another expansion of their polyvinyl chloride plant at Barry, South Wales.

**Bunawerke Hüls G.m.b.H.** are planning to extend their present production programme, which comprises thirteen grades of cold rubber, still further this year by expanding on production of carbon black rubber. Total capacity at present is 120,000 tons and thus represents the largest potential source of synthetic rubber in the OEEC area.

**The Shell Chemical Co. Ltd.** announce that their new ethylene unit at Carrington has just been placed on stream. This unit was built by Kellogg International Corp., London, and is designed ultimately to produce 55,000 long tons per annum of ethylene from a straight-run naphtha feed. The No. II unit at Carrington is the first built for Shell by Kelloggs in the United Kingdom.

**The Shell Oil Co. of Canada Ltd.** are to build a £7 million gas processing and sulphur manufacturing plant in Southern Alberta. It will produce 100 million cu ft of processed gas per day for export to the U.S.A. Also, 1000 tons of sulphur per day and about 4000 barrels of liquid products per day will be made.

**AEI Plastics (Aldridge) Ltd.** have added a new 8250 sq ft injection moulding shop to their Aldridge plant. All types of thermosetting and thermoplastic mouldings will be produced and productive capacity will be increased by 200-300 per cent.

**The Regent Oil Co. Ltd.** have opened their new fuel oil terminal at Queen Alexandra Dock, Cardiff. Costing £½ million and built in under a year, it will fulfil the dual function of providing the company with ocean storage in South Wales for fuel oils, and of replacing their present Light Oil Depot at Penarth by a modern installation with increased storage capacity and improved distribution facilities.

# IMPORTANT ANNOUNCEMENT

**ZWICKY LIMITED** have been appointed sole agents in the U.K. for the world-famous

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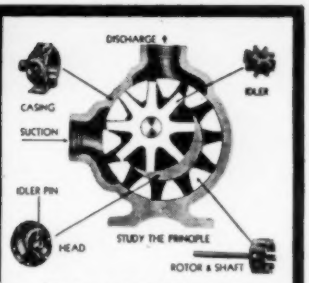


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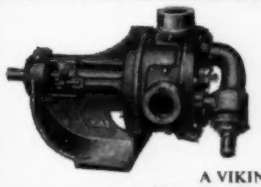

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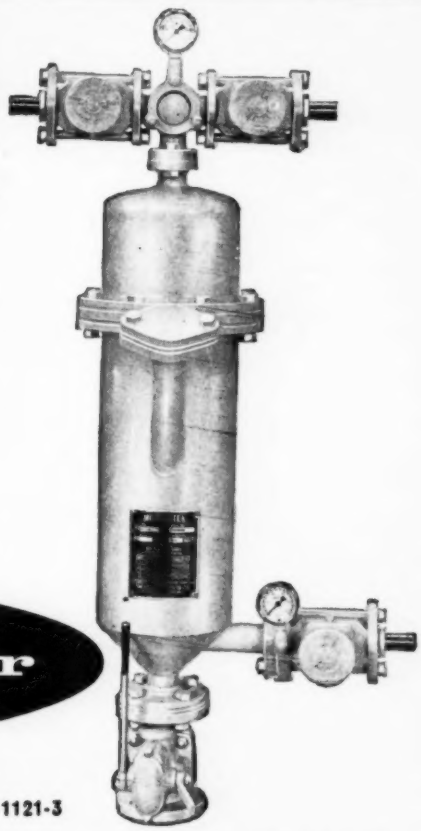

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### Construction Progress in The Netherlands

Good progress is being made on the Chemico gas reform plant under construction for MEKOG at Ijmuiden in The Netherlands. On this plant good use has been made of the Dutch waterways; the site located beside the North Sea Canal has provided easy access for some of the major equipment which has been floated to the plant limits or transported by barge. Heavy road transport has also played its part and some equipment has arrived on site by rail. The picture shows the carbon monoxide converter being taken from the canal to its location on the site.

## Company News

**Automatic Control Engineering Ltd.** is the name of the new wholly-owned subsidiary company of Constructors John Brown Ltd., formed from their Automatic Control Division. Services previously provided will be continued and developed by the new company.

**Amalgamated Oxides Ltd.**, of Dartford, announce that they have formed a joint subsidiary with Stolberger Zink A.G. of Aachen, having the title Stolberger Zincoli G.m.b.H. für Zinkstaub.

The new company will produce zinc dust for the paint industry, the demand for which is increasing in Common Market countries. Production will be by a newly erected plant at the German works.

**Dresser Industries Inc.**, of Texas, have acquired Podbielniak Inc., of Chicago, who manufacture centrifugal solvent extractors, laboratory distillation apparatus and packaged distillation plants. The British branch of the firm is Clark Bros. Co. Division of Dresser (G.B.) Ltd.

**Stockdale Engineering Ltd.**, of Cheshire, and the Mixing Equipment Co. Inc. (MIXCO), of New York, have formed a new company to be known as Lightnin Mixers Ltd. This company has taken over the well-known range of standard U.K. mixers, and will also market the complete line of "Lightnin" industrial mixers. The integrated range of fluid mixers now available will represent the most comprehensive line of this type of equipment obtainable anywhere.

## Contracts

**Mr. W. J. McBride**, a director of Edgar Allen & Co., has recently been on an eight-day visit to the U.S.S.R. In Moscow he lectured to top Russian executives and technicians on the design and manufacture of cement plant and kindred equipment. He was accompanied by DR. MERRYMAN, consultant to Edgar Allen's engineering division, who lectured on some physico-chemical aspects of cement manufacture.

**Whessoe Ltd.** have recently been awarded a contract by the Victoria Gas & Fuel Corp. for the design, supply and erection of one set of mild steel purifier boxes. They will have a capacity of 10 million cu ft per day, and will be incorporated in the West Melbourne gasworks (Australia).

**Lodge-Cotterill Ltd.** are to build blast furnace gas-cleaning plant for Dorman-Long (Steel) Ltd. for their Clay Lane Ironworks. The plant consists of two scrubber towers and three electrode filter units together with transformer-rectifier equipment. The value of the contract is about £300,000.

**At the British Trade Fair** being staged in Moscow next year, Fisons Ltd. will be demonstrating British industrial experience and know-how for the large potential market in the Soviet Union. During the past twelve months, Soviet technical missions have visited Fisons in Britain and return visits have been made. As well as technical experience, products immediately suitable for the Russian market, including pharmaceuticals and veterinary preparations, will be featured in the company exhibit.

**Head Wrightson Processes Ltd.** have been awarded a contract by the Central Electricity Generating Board worth nearly £½ million. They are to build a flue gas washing plant as part of the extension to Bankside power station. This plant is designed to remove more than 95 per cent of the sulphur from the oil-fired boiler flue gases.

## Recent Conferences

**Apart from the mammoth A.I.Ch.E.** event reported in the last issue of this journal, a number of other meetings of importance to chemical engineers have been held recently. The most important of these include a symposium on distillation held in Kiev, a conference entitled "Meeting on Chemistry" held under the auspices of the European Federation for Chemical Engineering in Helsinki, and a conference organised by the Institut Belge de Regulation et d'Automatisme in Brussels.

**At the Helsinki event** the papers given were devoted to research in chemistry, industrial and research co-operation for corrosion prevention, petrochemicals and industrial chemical developments. The first-mentioned group of papers covered such subjects as the investigation and observation of corrosion conditions (E. UUSITALO), stress corrosion in brass (EINAR MATTSSON), stainless steels for the sulphite cellulose industry (STIG BERG) and corrosion contact phenomena of metals (TENHO SNECK).

In the petrochemical field, polyolefins (B. ORSONI), alcohols by carbonylation (A. A. L. CHALLIS) and new resins for reinforced plastics (V. O. CLASSON) were the fields covered. The subjects of the final group of papers were electrochemical energy from secondary fuel elements (E. JUSTI), vacuum drying and degassing (M. BEYER), the refining of peat (A. SUNDGREN), graphite for chemical plant (A. HILLIARD) and the use of fluorocarbons in the chemical industry (F. BAUER).

**The application of analogue computers** for the control and design of chemical plant was the main subject of the 21 papers presented to the Belgian Institute. One author, J. I. ARCHIBALD, explained how the possible applications of a general-purpose analogue computer could be extended through the addition of a magnetic tape digital data storage, a feature which allows the simplification of some design problems through the ability to store partially complete calculations. Following upon this paper was another, given by A. W. O. FIRTH (Redifon Ltd.), on the optimisation of refinery processes.

## Personalities in the News

**Mr. J. McNicol Bruce** has been appointed Engineering Manager of the Blaw Knox Chemical Engineering Co. Ltd. For the past ten years MR. BRUCE has been employed with the Quickfit Group of Companies as Technical Director of Q.V.F. Ltd.

**Mr. G. U. Hopton**, recently Senior Research Chemist of the North Thames Gas Board, and, since August, an acting director, has been appointed the new Director of the Gas Council's London Research Station at the Fulham Laboratories.

**Dr. W. M. Campbell**, of Chalk River, Ontario, has been elected National Chairman of the Chemical Engineering Division, The Chemical Institute of Canada. DR. CAMPBELL is research director of the chemistry and metallurgy division, Atomic Energy of Canada Ltd.

**Mr. H. J. M. Stephenson**, previously a Director of W. H. Bailey & Sons, has accepted the appointment of Project Engineer in the Engineering Directorate of the U.K.A.E.A. at Production Group Headquarters, Risley.

**Mr. J. P. V. Woollam** was appointed deputy chairman of Simon-Carves Ltd. in December. He joined the firm in 1916 and has been a director since 1945.

**Profesor W. Hume-Rothery**, O.B.E., F.R.S., Isaac Wolfson Professor of Metallurgy in the University of Oxford, has been elected by the Council of the Institute of Metals to be an honorary member.

**Mr. C. J. Bullock** was appointed Chief Chemical Engineer of Bennett, Sons & Shears Ltd., of Leeds, during October 1960, and has now taken up this appointment.